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**FINAL REPORT
OF THE
REFRACTORY METALS SHEET ROLLING PANEL**

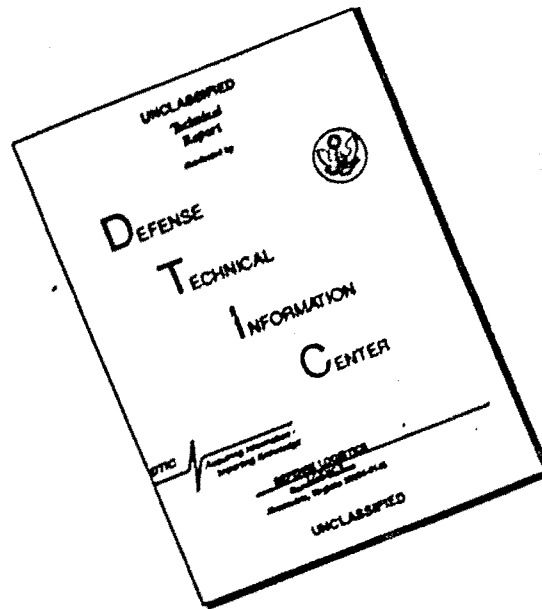


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Engineering Laboratories & Services
Missile & Space Systems Division
Douglas Aircraft Company, Inc.
Santa Monica, California

FINAL REPORT

of the

REFRACTORY METALS SHEET ROLLING PANEL

Prepared By The

MATERIALS ADVISORY BOARD

Division of Engineering
National Research Council

as a service of

The National Academy of Sciences

to the

Office of Defense Research and Engineering
Department of Defense

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This report completes a study undertaken by the Materials Advisory Board for the National Academy of Sciences in execution of work under ARPA Contract No. SD-118 between the Department of Defense and the National Academy of Sciences.

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CONTENTS

	<u>Page</u>
LIST OF TABLES	vi
LIST OF FIGURES	vii
ABSTRACT	viii
RECOMMENDATIONS	1
PANEL ACTIVITIES AND CONTRACT PROGRAM	4
Method of Operation	7
General Program Achievements	16
Summary of Status	26
Subpanel Functions	28
Introspection	34
Concluding Remarks	39
References	46
SUBPANEL SUMMARIES	49
Alloy Requirements & Selection Subpanel	50
Analysis Methods Subpanel	56
Coating Subpanel	63
Consolidation and Processing Subpanel	73
Ad Hoc Infab Subpanel	81
Joining Subpanel	85
Phase II Subpanel	89
Phase III Subpanel	95
Quality Specifications Subpanel	101
Standardization of Test Methods Subpanel	106
Steering Committee	109
Tubing Subpanel	111
APPENDIX I, Letter of Assignment	119
APPENDIX II, Alloy Requirements & Selection Subpanel Report	120
APPENDIX III, Evaluation of Candidate Refractory Metal Sheet Alloys by the U.S. Army Materials Research Agency . .	171

LIST OF TABLES

<u>Table</u>		<u>Page</u>
1	Subpanel Chairmen & Reports	8
2	Contracts of Primary Interest to the Refractory Metals Sheet Program	12
3	History of Alloys Identified for Production Development by Sheet Rolling Panel	17
4	Panel Recommendations in Current Program	40
5	Publications of the Refractory Metals Sheet Rolling Panel	44
6	Target Properties & Properties of Recommended Alloys (as presented)	54
7	Basic Required Properties for Comparative Evaluation of Structural Metallic Materials for Phase II	93
8	Targets for Refractory Metal Tubing Alloys	115
	<u>Appendix II</u>	
1	AIA Survey of Refractory Metal Requirements	129
2	Targets for Alloy Selection	133
3	Compliance of Molybdenum Alloy Sheet Candidates with Fabricable Molybdenum Alloy Target Properties	139
4	High Strength Molybdenum Alloy Candidates	141
5	Compliance of Columbium Sheet Alloy Candidates with Target Properties	143
6	Compliance of Columbium Alloy Candidates with Targets	147
7	Compliance of Fansteel and Haynes Stellite Columbium Sheet Alloys with RMSRP Targets for Fabricable Columbium Alloys	149
8	Compliance of Columbium Alloy Candidates with Fabricable Alloy Targets	153
9	Compliance of Columbium Alloy Candidates with MAB Fabricable Columbium Alloy Targets	157
10	Properties of Candidate Tantalum Alloys	159
11	Tantalum Alloy Candidates	162
12	Compliance of Tantalum Alloy Candidates with MAB Targets	164
13	High Strength Tungsten Alloy Candidates	166
14	Summary of Advanced Tungsten Alloy Development	168

LIST OF FIGURES

<u>Figure</u>		<u>Page</u>
1	Alternate Routes Investigated for Production of Sheet from a Molybdenum Alloy	15
2	Comparison of Properties Achieved to Targets	18
3	Curved Channel - 0.060 Inch TZM Molybdenum after Finish Forming at 300 ^o F in Sheridan-Gray, Double Acting, Hot Sizing Press	20
4	Corrugation - 0.016 Inch TZM Molybdenum Formed at Room Temperature	21
5	Dimpled Skin and Corrugation - 0.016 TZM Molybdenum Dimpled at 300 ^o F	22
6	Corrugated Test Panels from 0.020 Inch Tungsten Sheet . .	23
7	Deep Drawn Cups Made from 0.06 and 0.100 Inch RMSRP Tungsten Sheet	25

ABSTRACT

The genesis, method of operation, and accomplishments of the Refractory Metals Sheet Rolling Panel are described in the report of the main Panel, along with reflections on the conduct of such a program and recommendations for future activities in this field.

Summary reports of the eleven subpanels and one special ad hoc subpanel are included in the body of the report. Longer discussions of activities of the Subpanel on Alloy Requirements & Selection constitute an appendix to the report.

FINAL REPORT
REFRACTORY METALS SHEET ROLLING PANEL

RECOMMENDATIONS

Introduction

The refractory metal sheet rolling program was originally established by the Department of the Navy, Bureau of Naval Weapons, to identify the variables and causes responsible for variation in refractory metal sheet and to develop remedies for these difficulties. It was intended, in general, to develop a comprehensive technology for making high-quality, reproducible, widely usable material, with all the implications therein, responsive to the established requirements of weapons and vehicle designers. The program subsequently was expanded through the Department of Defense to include the other Services, and National Aeronautics and Space Administration and Atomic Energy Commission in a broadly based, integrated effort.

At this writing, six years later, the refractory metal sheet industry in the United States is a going business. In the last few years there has been available: a choice of strong alloys; wide and thin sheet produced to close tolerances; a background of property data and formability experience, and finally, sufficient production know-how to permit reasonable deliveries and realistic quotations. Several of the currently available alloys were unknown at the start of the program. It is believed that the sheet rolling program has made a significant contribution to this progress.

Although the coordinated program is well along and it is clear that the general objectives have been achieved, some of the contractor programs need to be completed. Other program recommendations should be implemented as outlined in the body of this report in Table 4.

With the ending of the formal program, the following recommendations are offered:

1. a. Collection and dissemination of Phase I and Phase II data should be accomplished for the columbium alloys FS-85, D-43, and Cb-752 and for the tantalum alloys T-222 and GE-473 in limited-scope programs as defined in Table 4. A single summary document should be prepared that will include all pertinent reliable property data (Phase II data) along with the mill-processing history (Phase I data) for each of these materials.

b. Advanced tungsten alloys of both the high-strength and room-temperature ductile classes have shown exceptional promise. After additional laboratory optimization has been completed, a selected tungsten alloy or alloys should be scaled up to the pilot level for demonstration of production feasibility and for determination of preliminary design data.
2. A coordinated coating program should be continued as described in the Coating Subpanel report, page 63; additional detailed recommendations will be found in this subpanel report.
3. A coordinated tubing program should continue as recommended in the Tubing Subpanel report, page 111.
4. A "working forum" or a standing "Refractory Metals Requirements & Selection Panel" (RMRSP), including Government, consumer, producer, and R&D groups, should be created to review progress regularly, in the area of refractory metal developments leading to all needed product forms. The "minutes" of such meetings should be available to all to provide maximum information for guidance of both industry in-house and Government programs. It has been proved that proper action will follow if objectives can be clarified and made known to those who must respond. It is deemed an essential feature of such activity that an "Alloy Selection Group" would impartially select specific compositions to be recommended for Government support for process developments. The Panel suggests that this approach

may, indeed, be appropriate for a wide variety of materials required in Government programs, particularly where there is critical need and a small market.

5. Some additional recommendations will be found at the end of several of each of the subpanel reports in this document.

PANEL ACTIVITIES AND CONTRACT PROGRAM

Formation of the Refractory Metals Sheet Rolling Panel

At the inception there was a need for refractory metal sheet for certain research and development vehicles or devices such as the X-20, ramjets, and solid rocket components plus, certainly, the knowledge that with the constantly upward trend in operating temperatures, requirements would be present if quality sheet of the proper alloys could be provided. At that time very few refractory metal alloys were available, surface and dimensional control was poor, and worst of all, product quality was extremely variable. This was the era when unalloyed molybdenum was beginning to be replaced by the Mo-0.5Ti alloy, Cb-1Zr was the columbium alloy, when there were no tantalum alloys, and no sizeable tungsten sheet.

A major quality problem was lack of uniformity. Variable formability and tendency to delaminate or crack during shearing and forming (Ref. 1) were persistent problems in attempted applications. These problems were most pronounced with Mo- and W-based materials. All of the refractory metals considered in this program, W, Ta, Mo, and Cb, are body-centered cubic metals and at least W, Mo, and Cb exhibit a ductile-brittle transition temperature as temperature is lowered. It is desired that this DBTT be below room temperature to facilitate handling and forming. Molybdenum sheet was found to have an extremely variable DBTT, usually above room temperature.

In the "Report of the Committee on Refractory Metals" (Ref. 2) in 1959, it was stated:

"The principal deterrent to the use of molybdenum sheet is its unreliability in mill products produced today, and the limited technology that has been developed to fabricate it into engineering structures. Quality variations, ranging from unacceptable to acceptable, are seriously retarding the final

development of this metal as an engineering material. Fabrication problems such as fusion welding and protective coating are also a deterrent to its application. The great potential molybdenum sheet metal has for extending present airframe and engine structural concepts to higher operating temperatures, thereby resulting in the simplest, lowest weight structure, cannot be taken advantage of until the metal can be reliably produced and fabricated.

"In addition to the required improvements in metallurgical behavior, a considerable effort is necessary to provide rolling equipment, furnaces, and supporting equipment. These must be capable of rolling large (36-inch wide), flat, thin (down to 0.010 inch) sheets, with thickness tolerances the same or one-half those for steel, of the Mo-0.5Ti alloy and advanced alloys for structural application in airframes and engines."
(page 146)

This report concluded with a strong recommendation that a molybdenum sheet rolling program be instituted.

Surface contamination was another persistent problem. Columbium and tantalum alloys are particularly prone to contamination from oxygen and nitrogen when heated, and some Mo alloys are also susceptible. Such contamination reduced bend ductility and formability. The lack of uniformity also affected mechanical properties, and as a result many designers felt that refractory metals were not ready to be specified. These difficulties with refractory metal sheet have been described in more detail in Reference 1.

In an attempt to confirm the existence of applications for refractory metals, the Aerospace Industries Association (then the Aircraft Industries Association) polled the members of its Aircraft Research and Testing Committee. The three questions asked were: Which metals were of interest; yearly quantities required; and the desired characteristics in the metal. Returns from eighteen companies showed strong interest in molybdenum, columbium, and tungsten (totaling 15,000, 12,500, and 10,000 pounds of mill products), and lesser interest in vanadium, rhenium, chromium, and tantalum. With hindsight, it would appear that the limited interest in

tantalum (a total of 660 pounds was the estimated requirement for the eighteen companies) was not justified.

Objective of the Program

In the words of the 1959 Molybdenum Panel quoted previously, the objective was to "develop the technical information necessary to transpose previous limited processing . . . into a high-quality production sheet product suitable for use in aircraft and missile manufacture, and to evaluate the resulting material." It was decided not to restrict the scope of molybdenum. In practice, nearly equal emphasis has been given to all four important metals, molybdenum, columbium, tantalum, and tungsten. The program was not one of research but one of identification and development of alloys of promise.

Method of Operation

The Refractory Metals Sheet Rolling Panel decided that the program should be divided into three phases for each alloy:

- Phase I - Development and documentation of a production practice for high-quality sheet and production of a quantity of sheet to demonstrate and establish quality and uniformity.
- Phase II - Measurement of preliminary design data for the "pedigreed" sheet from Phase I.
- Phase III - Establishment of limits of formability and definition of forming and joining procedures for sheet, followed by tests of fabricated structural elements. In some cases prototype aerospace vehicle or propulsion system components were to be designed, fabricated and evaluated.

During the tenure of the main panel, twelve subpanels were created (Table 1) to aid in guidance, to provide standards, or to survey the state of the art and recommend needed research. The activities of each has been summarized in the main body of the report following this description of panel activities; they are highlighted herein, as necessary, to illustrate their specific functions in the activity.

Alloy Selection

Of major importance was the decision as to which refractory metals or alloys should be fed into the program. This portion of the activity was the responsibility of the Subpanel on Alloy Requirements & Selection. This group has repeatedly surveyed the requirements for these materials by consulting the consumers and by referring to the product of the Aerospace Applications Requirements Panel of the Materials Advisory Board (Ref. 3).

TABLE 1

Subpanel Chairmen & Reports

<u>Subpanel</u>	<u>Chairman</u>	<u>Publication</u>
Alloy Requirements & Selection	Dr. Robert I. Jaffee	----
Consolidation and Processing	Dr. Wm. Rostoker	MAB-179-M
Joining	Dr. Wm. Rostoker	MAB-171-M
Quality Specification	Mr. John T. Stacy	MAB-184-M MAB-190-M MAB-196-M
Phase II Guidance	Mr. G. M. Ault	----
Phase III Guidance	Mr. Roger A. Perkins	----
Test Methods	Mr. L. M. Raring	MAB-176-M MAB-192-M MAB-216-M
Analysis Methods	Dr. L. L. Scigle	MAB-178-M MAB-217-M
Coating	Mr. J. J. Gangler	MAB-181-M MAB-189-M MAB-201-M MAB-210-M
Tubing	Mr. Wm. Thurber	MAB-208-M
Steering	Mr. G. M. Ault	MAB-172-M MAB-188-M
Infab (Ad Hoc)	Mr. D. Goldberg	MAB-99-LM MAB-100-LM

They conducted, at the initiation of the program, a survey to learn the status of refractory metal alloy development in this country. Based upon these surveys, they decided it was desirable to set target properties for six specific classes of alloys:

1. Fabricable molybdenum
2. High-strength molybdenum
3. Fabricable high-strength columbium (originally separated into two classes)
4. Tantalum
5. Unalloyed or dilute tungsten
6. High-strength tungsten

The targets are described in the table on page 133. These targets served two purposes: (1) they provided the industry with specific objectives permitting them to focus their efforts for alloy development, (2) they listed specific test data that should be obtained to permit valid comparisons to be made. The stimulus for response by the industry was the opportunity for Panel endorsement and for Government support for Phases I, II, and III for the selected alloys.

An example of the philosophy in creating the targets can be seen by comparing the "fabricable" and "high-strength" molybdenum classes. The significant difference appears in high-temperature strength and room-temperature ductility requirements. The high-strength alloy was to have a comparable strength but at a 400°F higher temperature. This strength was to be obtained at a sacrifice in room-temperature ductility and ductile-brittle transition temperature.

An important point of philosophy can be illustrated for columbium alloys. It was stated (by the targets) that, to be of interest, columbium alloys must retain a major attractive characteristic of columbium -- good ductility in the welded condition at room temperature. Molybdenum alloys having high strength but lacking weld ductility were already available.

The targets were submitted to the industry and candidate alloys were screened. Following selection of candidates, the Government agencies could fund development through the three phases as deemed necessary.

The making of reliable comparisons of alloys requires dependable and comparable data. An important role was played by the Army Materials Research Agency which established a laboratory facility specifically for providing a uniform evaluation of candidate alloys. The mechanical properties of most alloys, as measured at AMRA, checked producers' results rather well with only occasional controversies. This reflects continued coordination and cooperation of participants in improving and standardizing testing techniques during the course of the program.

Alloy selection has been an intensive process spanning several years. It required an estimation of future (and unknown) requirements, a knowledge of present capabilities, and a need to balance producibility against high properties. Those involved in the program were impressed with the manner in which industry responded to the challenge. Once clear objectives had been established, producers, whether under contract or not, made rapid progress so that within a few years several alloys in each class were available for selection. It is significant to note that at this date the target properties have been achieved for all classes except high-strength molybdenum.

As will be appreciated, it is a long road from a laboratory sample to commercial availability of large sheet with good surface and flatness and close tolerances and reproducible properties. Largely, this is what the refractory metal sheet rolling program was all about. During scaleup, the composition may change, mechanical properties do not always hold up, segregation is often a problem and so forth. Nevertheless, in this period, we have seen the process development accomplished for alloys of molybdenum, columbium, and unalloyed tungsten, with tantalum alloys not far behind.

Contractor Programs

The alloys and contracts of all three phases of the program are indicated in Table 2. The first column lists by classes, the alloys that have been selected for scale-up by the subpanel, and the other columns list the contractors. Additional contracts have been recommended as will be described in a later section.

It is beyond the scope of this summary to detail the technical details of the 14 or 15 contracts of the program. The contractors regularly documented their progress, however, and DMIC has issued reports (Ref. 4) summarizing all contractor achievements. A report summarizing all contractor progress to date will be released by DMIC in 1966. Highlights will be described later herein.

Molybdenum

The fabricable molybdenum alloys Mo-0.5Ti and TZM (Mo-0.5Ti-0.08Zr-0.03C) were supported in Phase I and this phase is complete. TZM is proceeding through the subsequent two phases.

The status of development of high-strength molybdenum alloys was reviewed April 1963 and TZM (Mo-1.25Ti-0.3Zr-0.15C) was identified to be of interest. Because of a lack of a definite requirement for such alloys and because development of production processes would be costly, development scale-up has not been recommended at this time.

Tungsten

In the case of unalloyed tungsten, two production routes have been investigated, powder metallurgy and arc cast, in Phase I. The

TABLE 2

Contracts of Primary Interest to the Refractory Metals Sheet Program

	<u>Pilot</u>	<u>Phase I</u>	<u>Phase II</u>	<u>Phase III</u>
<u>Fabricable Mo</u>				
Mo-0.5Ti TZM		Universal Cyclops(BW)	Southern Res.(BW)	McDonnell(BW)
<u>Unalloyed W</u>				
Powder Met.		Fansteel(BW)	Southern Res.(BW)	Solar and Super-Temp(BW)
Arc Cast		Universal Cyclops(AF)		
<u>Tantalum</u>				
30Cb-7.5V T-222 GE-470	Westinghouse(BW) GE In-House Funded	Wah Chang(AF)		
<u>Fabricable (and weldable) Cb</u>				
D-43(X110)		Crucible - duPont(AF)		
B-66, FS-85 Cb-752	Fansteel and Westinghouse(BW) Haynes-Stellite(AF)			
<u>Foil Contract</u>	Metals & Controls (AF)	duPont(AF)		

Sponsors: PW - Bureau of Naval Weapons
AF - Air Force Materials Laboratory

powder metallurgy material is being investigated in Phase II and III programs also. The Panel has not endorsed support of unalloyed arc-cast tungsten beyond Phase I although some evaluation of Phase I material would be expected. Phase I is complete for the powder metallurgy tungsten, but not for the arc-cast material. Phases II and III are nearing completion for the powder metallurgy material.

Tantalum

In tantalum alloys the Panel has recommended no additional support for the Ta-30Cb-7.5V alloy but has urged continued effort on the T-222 alloy (Ta-9.6W-2.4Hf-0.01C) developed by Westinghouse under BuWeps support and the GE-473 alloy (Ta-7W-3Re) developed by the General Electric Company. These latter alloys have been through pilot programs (GE-473 with company funds) to demonstrate their producibility and outstanding properties. Because establishment of quality and uniformity in production is deemed straightforward and simple, a modest Phase I program, has been recommended. This would be followed by a Phase II program to determine properties of the pedigreed sheet. Phase III has not been recommended.

Columbium

For columbium alloys, the Panel recommended FS-85 (Cb-28Ta-10W-1Zr) and B-66 (Cb-5Mo-5V-1Zr) for "pilot" support with two producers investigating both alloys. From the results of those programs, FS-85 was recommended for further development in a program similar to that recommended for T-222 and GE-473 (see above). The Air Force Phase I sheet program on D-43 (Cb-10W-1Zr-0.1C) was endorsed and the panel regularly reviewed the Air Force "pilot" program on Cb-752 (Cb-10W-2.5Zr). Although formal Phase II and Phase III programs have not been conducted on D-43 and Cb-752,

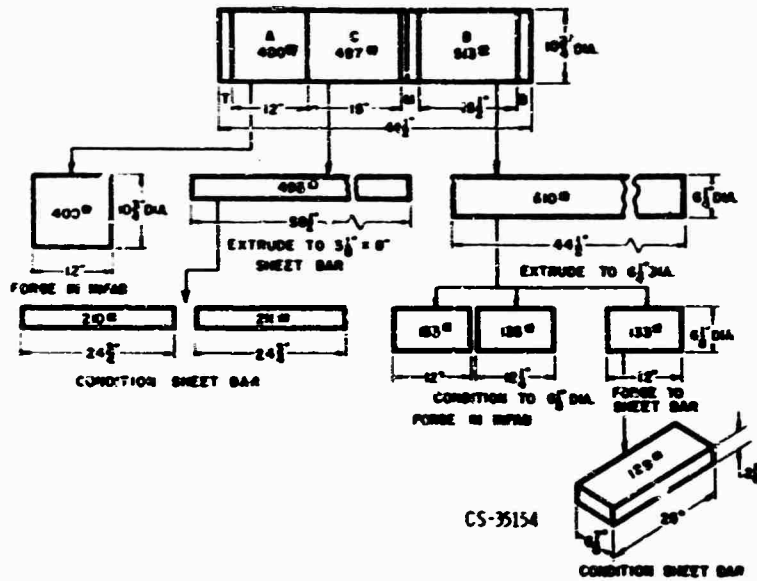
material has been distributed to those who may obtain such data. The Panel has recommended that all of this information for each alloy be collected and published by DMIC.

Foil

The last line on the table describes work on production of refractory metal foil of tungsten and certain columbium and tantalum alloys. Foil to 0.002-inch thickness has been produced in some of these materials.

The progress of the contractors has been gratifying from the technical viewpoint. Without Government support the contractor generally cannot afford to explore alternate routes to achieve a high-quality sheet product in the face of an uncertain market when material costs may be, say, 50 dollars a pound. Figure 1(a) and (b) show the many alternate processing routes explored for one material in the Government-funded program. Given this opportunity, the producer has explored enough alternate processes to devise one capable of achieving a consistent reproducible high-quality product.

INGOT TO SHEET BAR



SHEET BAR TO SHEET

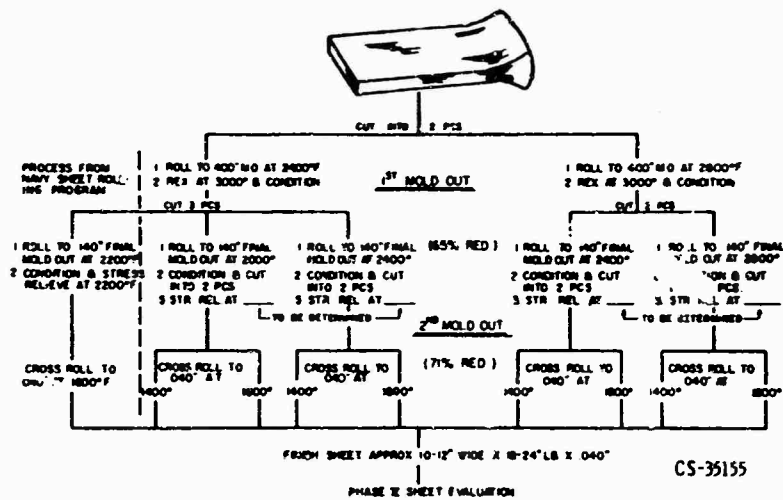


Fig. 1 - Alternate Routes Investigated for Production of Sheet from a Molybdenum Alloy

General Program Achievements

To highlight the progress under the program, we may compare in Table 3 the current status of the alloys for high-quality sheet with their status when the program began in November 1959. This table shows that several of the alloys which have advanced within the time period of the program to a point where sheet can be produced in large sizes with good quality and uniformity were unknown at the start of the program.

Another point of interest is to compare the properties of the selected materials that have been investigated in the scaleup program with the more significant targets of strength, ductility, and weldability, Figure 2. For each of four classes, the bar graphs compare the high-temperature tensile strengths of the selected alloys to the targets (shaded bars). The tabular data at the top of the figure compare the ductile-brittle transition temperatures with targets and also compare the ductility at room temperature after welding with the target.

For the fabricable molybdenum class, the selected alloy, TZM, exceeded the strength targets at 2000° and 2400°F but of more significance is the ductile-brittle transition temperature comparison. Early in this summary, problems in molybdenum were described with emphasis on the fact that the only consistent feature of the DBTT was that it was almost always above room temperature. As a result of the program, attention has been focused on an alloy, TZM, having much better strength that now consistently demonstrates a DBTT below minus 60°F. Room temperature weld ductility was, of course, not sought in molybdenum and not achieved.

Unalloyed tungsten was pursued for use at very high temperatures and met the targets for tensile strength at 3000° and 3500°F. Capability of producing large sheet with good quality and uniformity in flatness and in gage control was sought and generally achieved. Ductility at room

TABLE 3

History of Alloys Identified for Production
Development by Sheet Rolling Panel

<u>Alloy Class</u>	<u>Status November 1959</u>	<u>Status - 1965</u>
<u>Fabricable molybdenum</u>		
Mo-1/2Ti	Large sheet poor quality	Completed production program (24 x 72" sheet)
TZM (Mo-0.5Ti-0.1Zr-0.03C)	Small sheet	Completed production program (24 x 72" sheet)
<u>Tungsten</u>		
Unalloyed	Lab. size sheet	Completed production program (18 x 48" sheet)
<u>Fabricable & weldable columbium</u>		
D-43 (Cb-10W-1Zr-0.1C)	Unknown	Completed production program (24" wide)
Cb-752 (Cb-10W-2.5Zr)	Unknown	Completed production program (24" wide)
FS-85 (Cb-28Ta-10W-1Zr)	Unknown	Completed pilot production (13" wide)
<u>Tantalum</u>		
T-222 (Ta-10W-2.5Zr-0.01C)	Unknown	Completed pilot production
GE-473 (Ta-7W-3Re) ^(a)	Unknown	Completed pilot production

^(a) Development funded by G. E.

COMPARISON OF PROPERTIES ACHIEVED TO TARGETS

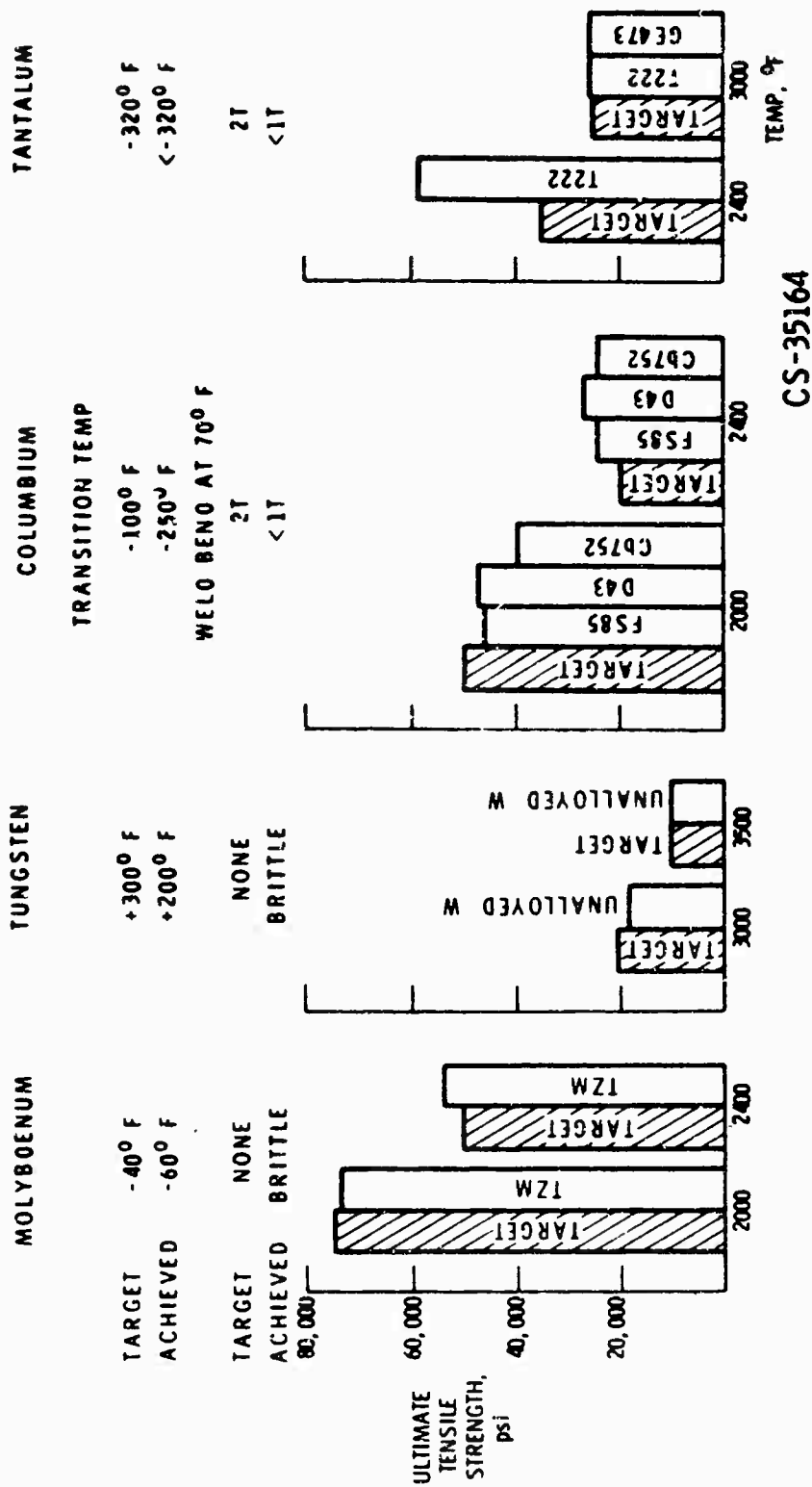


FIGURE 2

temperature was not expected. However, recent presentations to the Alloy Requirements and Selection Subpanel indicate that room-temperature ductile tungsten alloys may be achieved when moderate rhenium additions (approx. 5%) are present.

Columbium alloys were sought having good strength but with major emphasis on the requirement that they should be ductile when welded. (Otherwise they would have little advantage over molybdenum alloys.) The resultant alloys described in Figure 2 are of high strength, have a DBTT of lower than minus 250°F, and FS-85 and D-43 after being welded will survive a bend over 2T radius at room temperature.

Tantalum alloys were sought having a higher use temperature than columbium alloys and with even better ductilities. These have been achieved.

Another indication of progress is to note the formability of the sheet product of the Phase I programs in contrast to the earlier status presented in the first paragraphs of this summary. The determination of formability and formability limits has been the responsibility of Phase III contractors with objectives and progress guided by the Phase III Subpanel. These studies are underway for the molybdenum alloy TZM at McDonnell (e.g., Ref. 5) and for tungsten at Solar (e.g., Ref. 6) and at Super-Temp (e.g., Ref. 7) under sponsorship of the Bureau of Naval Weapons.

Formed parts of TZM are shown in three figures: a curved channel, Figure 3, corrugations formed at room temperature, Figure 4, and a dimpled corrugation, Figure 5. All of these figures are from the contract of Ref. 5.

Formed parts from powder metallurgy tungsten are shown in Figure 6(a) and (b) and show the apparatus for hot forming a corrugated test

CURVED CHANNEL - 0.060 INCH TZM MOLYBDENUM AFTER FINISH FORMING
AT 300° F IN SHERIDAN-GRAY, DOUBLE ACTING, HOT SIZING PRESS

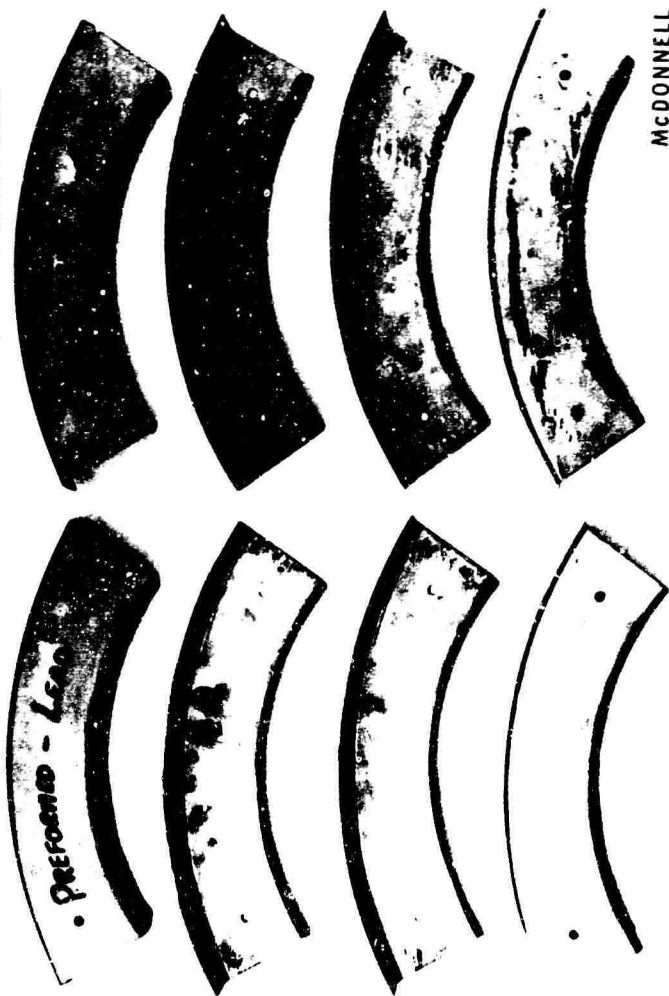


FIGURE 3

CORRUGATION - 0.016 INCH TZM MOLYBDENUM
FORMED AT ROOM TEMPERATURE

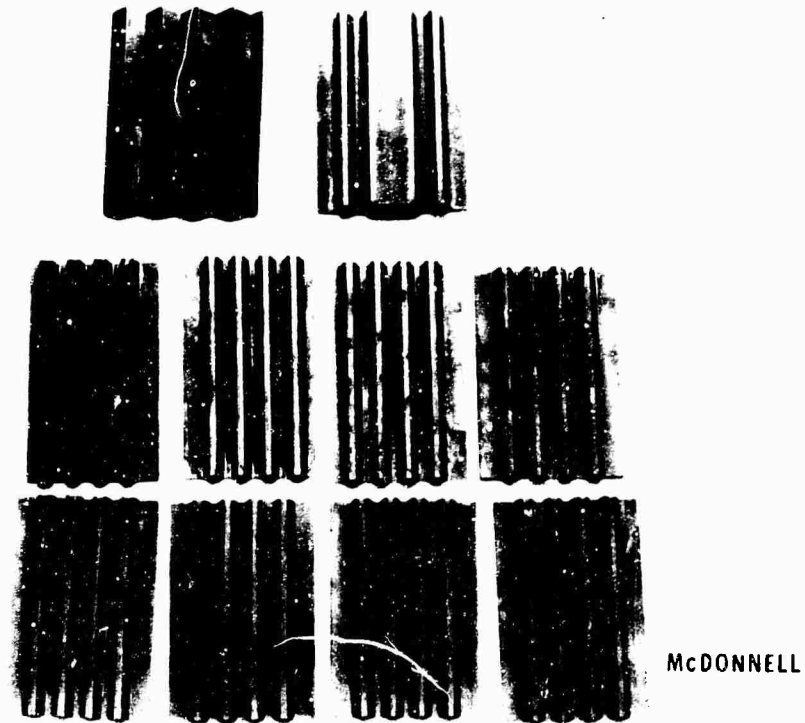


FIGURE 4

DIMPLED SKIN AND CORRUGATION - 0.016 TZM
MOLYBDENUM DIMPLED AT 300° F
Hole Diameter - 0.038

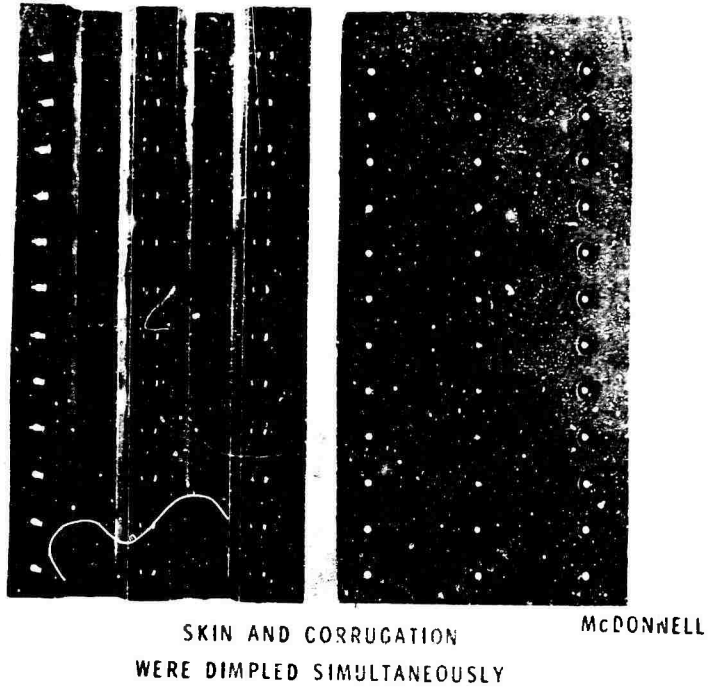
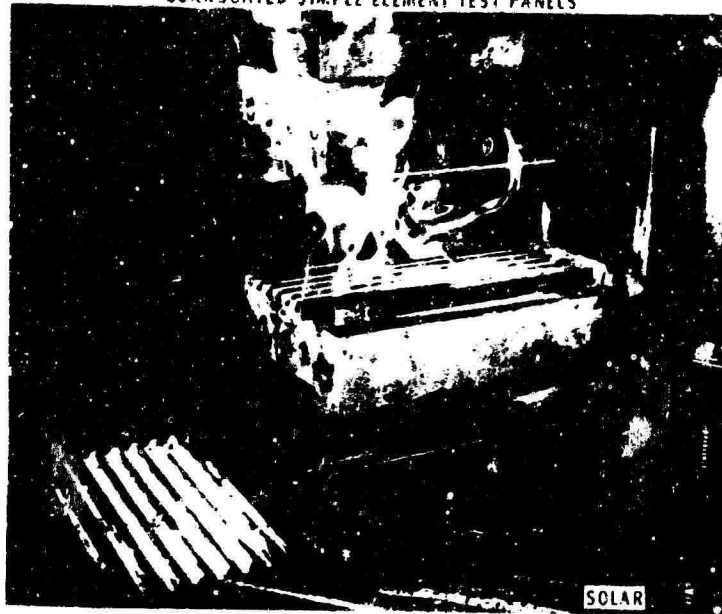


FIGURE 5

(a) CLOSEUP OF HOT CORRUGATING DIE SETUP FOR FORMING
CORRUGATED SIMPLE ELEMENT TEST PANELS



(b) Fabricated panels

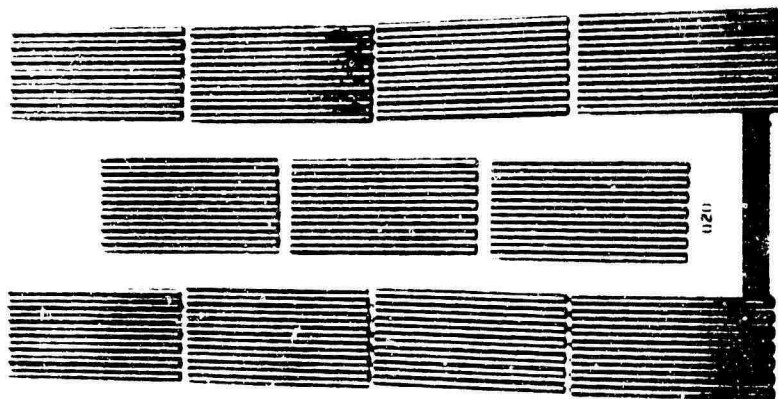


Fig. 6 - Corrugated Test Panels from 0.020-inch
Tungsten Sheet

panel, Figure 6(a), and finish pieces, Figure 6(b). Deep drawn cups that were drawn at about 1650°F are shown in Figure 7. These figures are from the contract of Ref. 7. Clearly, it has been shown that proper procedures can produce quality parts of these alloys. This was expected. An important product of the Phase III contractors will be the provision of guidelines for the forming of these materials.

A particularly important contribution of the panel activity has been the output of certain of the subpanels. The Test Methods Subpanel has provided guidelines for testing of refractory metals where none existed before. The Coating Subpanel similarly provided needed recommendations for standard tests for coated refractory metals. The Quality Specifications Subpanel has provided targets for refractory metal sheet quality and outlined sheet sampling methods. All are being widely used. The Analysis Methods Subpanel has guided round robins for measurement of capability of analysis methods in refractory metal alloys. Several of the panels have recommended needed research that has been supported by the services.

DEEP DRAWN CUPS MADE FROM 0.06 AND 0.106 INCH
RMSRP TUNGSTEN SHEET

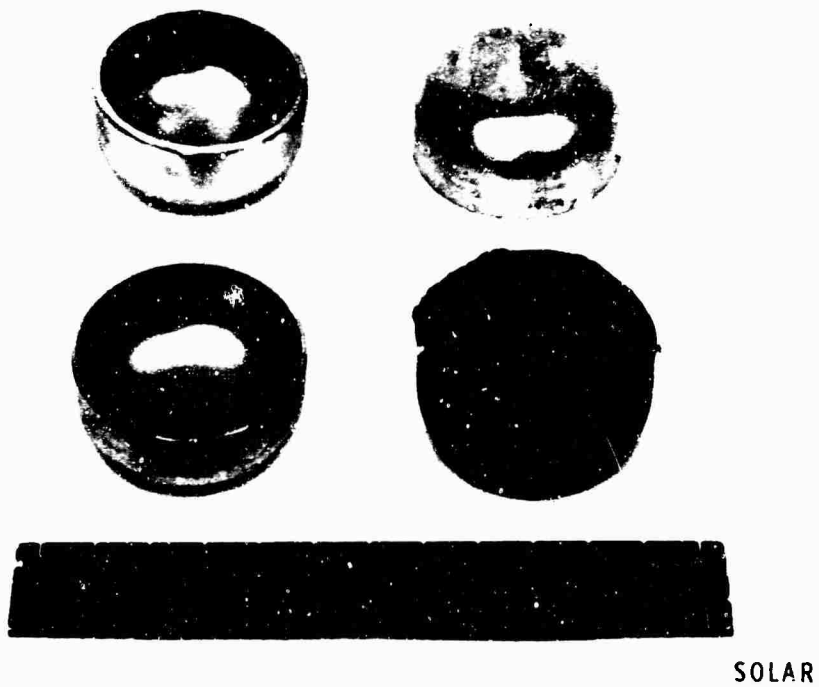


FIGURE 7

Summary of Status

The following alloys have been involved in the refractory metal sheet rolling program:

<u>Alloy Base</u>	<u>Involved in RMSRP</u>
Molybdenum	Mo-0.5Ti, TZM (Mo-0.5Ti-0.08Zr-0.03C)
Columbite	FS-85 (Cb-28Ta-10W-1Zr) D-43 (Cb-10W-1Zr-0.1C) Cb-752 (Cb-10W-2.5Zr) B-66 (Cb-5Mo-5V-1Zr)
Tantalum	T-111 (Ta-8W-2Hf) T-222 (Ta-10W-2.5Hf-0.01C) 30Cb-7.5V (Ta-30Cb-7.5V)
Tungsten	Unalloyed

Available Quality

It has been demonstrated that large sheets of high-strength refractory metal alloys can be produced to meet consistently the following specifications:

Thickness tolerance	1/2 AMS 2242
Edge camber	3/16" max. in 8'
Flatness	4% (half-chord) to 6% in the case of thin-gage tungsten
UTS	+7% about the mean (+5% in one lot)
YS	+15% about the mean (+8% in one lot)
2000°F UTS	+10% about the mean
2000°F YS	+15% about the mean

Transverse bend transition	not more than 50°F in one sheet;
temperature variation	not more than 50°F in one lot
	(150°F for tungsten)

Transverse and longitudinal bend transition temperature variation
not more than 100°F in one sheet or lot (except 150°-200°F in the
case of tungsten)

Property Data

The mechanical properties (strength (both room and elevated temperature), creep rupture, recrystallization temperature, transition temperature, and bend ductility) have been obtained using standardized test techniques on "pedigreed" sheet on molybdenum alloys (TZM and Mo-1/2Ti) and unalloyed tungsten. In addition, considerable but less detailed data are available on FS-85, D-43, Cb-752, and B-66 columbium alloys, and on several tantalum alloys.

Formability Limits

The producibility of hardware made from tungsten and molybdenum (TZM) has been demonstrated by work carried on at Solar, Super-Temp, and McDonnell. The types of fabricating operations, forming limits, temperature and strain rate restrictions, etc., have been established.

Production Know-How

A sufficient number of production variables have been explored so that optimized reproducible methods have been established. Thousands of pounds of several of the alloys have been produced, lending confidence in the chosen methods.

These accomplishments are the result of industry-government cooperation generally within the framework of a coordinated program guided by the Refractory Metals Sheet Rolling Panel of the Materials Advisory Board.

Subpanel Functions

The eleven subpanels were indicated in Table 1. Their accomplishments and recommendations are described in Part II of this volume and highlighted in the following paragraphs. The role of the Alloy Requirements and Selection Subpanel has been described previously.

Steering

This group, as the name implies, channeled the main panel deliberations toward particular problem areas and planned the agenda for the main panel meetings.

Consolidation and Processing

Consolidation and Processing are the focus of the Phase I activity. A subpanel was created early in the program to review this problem area, looking separately at the problems of consolidation, hot working, and cold working. Specific research and development projects were recommended (MAB-179-M, Ref. 8) which could lead to improvements in quality, recovery, and cost. It has been most gratifying that this report has been useful to the responsible representatives of government agencies who have initiated research and development in most of the recommended areas.

Joining

The Joining Subpanel conducted a similar state-of-the-art study (MAB-171-M, Ref. 9) in its area.

Quality Specifications

The Quality Specifications Subpanel provided guidance on targets for quality (flatness, gage, uniformity, etc.) in Phase I production programs

and provided a report describing methods for sampling of sheet and measurement of quality to determine whether these targets had been met (Ref. 10). After the tungsten sheet had been produced, they evaluated the quality and provided reports on recommended quality specifications for tungsten plate (Ref. 11) and tungsten sheet (Ref. 12).

Phase II Guidance

The subpanel outlined the data to be obtained in the Phase II program. The decision as to which data were to be obtained was based upon recommendations of the Aerospace Industries Association upon which the judgments of the panel members were superimposed.

Phase III Guidance

The Phase III Subpanel is a guidance group that outlines specific objectives and a general method of approach for those programs that have as their purpose to determine formability and to fabricate test components from the "pedigreed" sheet.

Test Methods

The Test Methods Subpanel has outlined recommended testing methods (Ref. 13) for refractory metal sheet. Guidelines for evaluation of these highly reactive and expensive materials have not been available previously and thus many laboratories are following the recommendations of the Subpanel. The test procedures were developed following ASTM guidelines for other materials as closely as practical. Methods are described for:

Tensile Tests at room and elevated temperatures	Shear
Compression	Bend
Notch tensile	Bearing

Stress rupture

Creep

Weld evaluation

Delamination

Recrystallization

Fatigue

Thermal conductivity

Thermal expansion

Specific heat

Modulus of elasticity

Analysis Methods

Another major problem area in refractory metals has been to achieve high precision and accuracy in chemical analysis. Three groups are pursuing this area, the Subpanel on Analysis Methods of this program, the ASTM, and a committee under the Advisory Group for Aeronautical Research and Development of NATO (AGARD). The Subpanel dealt specifically with the important materials (usually alloys) of this program. The methods used in this country were surveyed and the state of the art was assessed (Ref. 14). Based on this survey it was decided that round robins were needed to improve the quality of the analysis and to better define the areas needing additional research. The round robins are directed specifically at alloys of importance in the sheet rolling program and include a material from each of the four refractory metals. The alloys are:

Molybdenum alloy	TZM; Mo-0.5Ti-0.08Zr
Columbium alloy	FS-85; Cb-27Ta-10W-0.7Zr
Tantalum alloy	T-111 [*] ; Ta-8W-2Hf
Tungsten	Unalloyed

The Bureau of Naval Weapons contracted with the Bureau of Mines Laboratory in Albany, Oregon, to produce homogeneous standard "reference materials" of each alloy for the round robin. The results of the round robins will be published by DMIC as Report 220, and by MAB (Ref. 15). This summary

* T-111 is an early version of T-222. Its analysis problems should be similar to those of T-222.

will indicate the levels of precision achieved and the methods used for both interstitial elements and major alloying elements.

The National Bureau of Standards has agreed to provide a repository for the remaining "reference material" of each alloy, to distribute this material to others who wish to compare their methods to those of the original round robin, and, finally, to publish summaries of any such additional analyses.

Coating

Many of the important applications requiring the high-temperature strength of the refractory metals also involve the use of these metals in an air environment. Unfortunately, none may be used without coating and the provision of satisfactory coatings has been possible to date only for some limited (though important) short-time and/or relatively modest temperature situations. A major responsibility has fallen upon the Coating Subpanel to investigate this problem area.

The first act was to evaluate the state of the art with the aid of an intensive questionnaire, circulated to all organizations active in research and development or production of refractory metal coatings. These results were summarized in MAB-181-M (Ref. 16). It was immediately obvious that a major difficulty in assessing coating progress was that similar tests were not being run for various coatings -- test results could not be compared. The Subpanel devised and recommended standard tests for coating evaluation (MAB-189-M, Ref. 17). Currently, coating specialists are utilizing these recommended evaluation procedures for evaluating their coatings when applied to certain refractory metals of the sheet rolling program. The Subpanel has reviewed this progress and recommended further studies for sponsorship (Ref. 18).

The Subpanel has repeatedly emphasized that a major problem with coatings is the interaction upon the properties of the base metal (with the

effect usually detrimental). The coating must be tailored to the base metal composition (and vice versa) the coated refractory metal should be treated as a new composite material.

It is recommended that this coordinated coating activity continue beyond the lifetime of the main panel.

Tubing

In the judgment of the Panel, one of the more important product forms for the use of refractory metals will be that of tubing. Of particular significance is that the characteristics required of a material for high-quality tubing are those of a material for high-quality sheet. The sheet program has attempted to identify and to develop those refractory metal compositions having the highest strength concomitant with a capability of being formed into high-quality sheeting. Compositions capable of being formed into high-quality sheeting are also the best candidates for fabrication into high-quality tubing. For space power systems, the tubing should be weldable and should be ductile after welding. These same requirements were imposed upon tantalum and columbium alloys in this sheet rolling program. Finally, the high-strength tantalum and columbium alloys contain zirconium or hafnium. It has been found that these same additions provide resistance to corrosion by alkali metals proposed for Rankine systems. Thus, the alloys of the sheet program are being investigated for high-strength refractory metal tubing. In many of the applications for tubing, e.g., Rankine and Brayton cycle space power systems, coatings will not be required.

The Tubing Subpanel has published a state-of-the-art review covering, for tubing: requirements, methods of manufacture and our capability in manufacture, methods of nondestructive testing for small-diameter thin-wall tubing, and experience in production of such tubing from refractory

metal alloys of interest. (Ref. 19). Based upon the state-of-the-art review, specific recommendations are offered for future research and development.

It is recommended that the Tubing Subpanel activity continue beyond the life of the main panel.

Ad Hoc Infab

Although operations in Infab had been carried on since 1961, uncertainty existed as to the actual inertness of the atmosphere, and the metal processing functions which the facility might best perform. Because of the possible importance in molybdenum alloy sheet production, an ad hoc committee reviewed the problem. A specific program to measure contamination was recommended. Two reports of the Subpanel discussed technical and economic advantages of processing in Infab.

Introspection

Because this operation may be imitated in the future, it is important to review the method of operation in retrospect, while the experience is still fresh, to illuminate the shortcomings and difficulties as well as the good features. As pointed out in the section "General Program Achievements", the industry developed the capability of making sheet of the required quality of the needed alloys with minimized duplication and within a relatively short period of time. This measure would indicate that the operation, as a whole, was a success. Though the program is not complete, the objectives are being accomplished. Favorable comment can be made about the operation from the point of view of flexibility, the manner in which it was not tied to a weapon system, or to a service, the manner in which producers and users were brought together, and to the effectiveness in which the requirements for refractory metals and the target properties were publicized. The operation also was quite economic in that coordination between services was excellent. The outstanding manner in which the Phase III Subpanel helped the fabrication contractors in clarifying and maintaining the primary objectives is an example of the operation at its best. It is the Panel's opinion there should be no hesitance about recommending this type of operation in the future where an important class of materials will be needed, where the technical problems are severe, where interaction between producer and user is important, and where the time scale for development must be compressed.

The general format used (originally developed for the titanium sheet rolling program) is deemed to be sound and important to success. This format consists of 1) setting targets for alloy selection based upon a consideration of requirements and potential capability; 2) selection of alloys, from all candidates offered, for scaleup development; 3) providing technical guidance for the three phases (development of production capability, design data, and evaluation of fabricability), and 4) continuous review of contracted programs to insure compliance with objectives.

There were, however, some problems and shortcomings. The criticisms relate more to operating details within the format and not a quarrel with the format. Specific comments follow:

Success in Gaging Requirements

The point is made in another part of this report that frequent discussions covering refractory metal sheet requirements were held. At an early stage, a Table of Alloy Target Properties was issued, based in part on anticipated requirements. The impression may have been given that an accurate estimate of quantity requirements was at hand. Our very preoccupation with the problem may have led some producers to assume that a substantial market was present or imminent. This estimation of technical requirements should not be confused with an estimate of a market. It is possible that a confusion of this kind did occur on the part of some producers. While the Panel cannot be blamed for promising a market, possibly they should have done more in differentiating between technical requirements and quantity requirements. Looking back, it appears that the property targets established five years ago as required and attainable were a remarkably good projection.

Phase I

Coordination and Monitoring

Phase I involved development and documentation of a production practice for high-quality sheet of the selected alloys and as such was the cornerstone of the contractor programs. At the beginning, the Panel spent an inordinate amount of time going over the details of performance of Phase I contractors. It was learned that it is imperative to induce the contractors to confine their presentation to the major problem areas. Being involved in the details of their problems, a few contractors tended to present every such detail to the Panel, inundating them in tables and charts.

The very variety of difficulties which the contractors got into led the Panel to spend more time on this phase than was warranted.

The preoccupation with reviewing contractor performance in detail, however, was not merely due to curiosity. It is important that the major problems be identified and attacked, but it is not always obvious in advance what these major problems will be. Many difficulties arise only after work is well underway. The time spent with contractors, therefore, was a direct outcome of the recognition of unexpected roadblocks, for which recommended courses of action were needed. Some examples of these, from the early years of the program are:

The unsatisfactory alloying of molybdenum in powder metallurgy processing. Program returned to a research phase.

Problems with utilizing Infab, including limitations of impactor capacity, feeding into rolls of the mill, and initial uncertainties in measuring the contaminants in the atmosphere.

The difficulty in flattening roll-formed tungsten cylinders to make sheet.

Two alternatives are possible:

Assign specific Panel members responsibility for certain contracts and ask them to report (along with the contractors) to the main panel. This has been tried recently with only modest success.

Provide a small guidance group for the Phase I contract program in the same manner as was so successful for Phase III activities. This is the preferred solution.

Investigation of Process Variables

The mechanical properties of refractory metals are influenced greatly by cold work and by dispersed-phase chemistry and morphology which are controlled by processing variables. Clearly the control of processing

variables controls quality and reproducibility. This was the basis for the Phase I programs and studies were conducted as necessary to develop the needed quality. It is thought that more effort by trained "researchers" to explore this opportunity earlier in the program would have produced additional knowledge that could have been factored into the program.

Scope

Early in the program, the Panel became involved in review of "research-type" contracts seeking new methods of purifying refractory metals. It soon became obvious that results of such research, even if successful, could not be reduced to practice within the time scope of the program. Such diversions should be avoided in a development program such as this.

Improvement of "Statement of Work" for Contracts

The "monitoring" of contracts was made considerably more cumbersome because the work statements occasionally did not conform to what the Panel felt should be done, usually as a result of difficulties in timing (the long-time cycle in agency procurement had required initiation of contracts prior to Panel consideration). Nearly always the Panel was faced with a fait accompli, often with contracts on which the work was well underway. The agency contract manager was always receptive and responsive to the recommendations of the Panel; but it was generally difficult, and sometimes impossible, to correct situations when they became apparent because of the contract wording by which the contractor was committed to other tasks. Strong expressions of dissatisfaction with this situation were made as early as the August 1960 meeting. In the future, every effort should be made to have the recommendations of the Panel prior to initiating the requests for proposals.

Panel Membership

Contract review may have worked out better had representatives of producers been on the Panel. The philosophy adopted was to assemble a group of design and metallurgical experts from airframe and power plant companies, and from individuals with competence in powder metallurgy and forging, as well as non-industrial metallurgists. No one from among the producers of refractory metal sheet was included. It was felt that sufficient competence on production problems was present, and that to ask one producer to review the work of another would have been an intolerable situation. After about a year, a regular practice was adopted of inviting (usually two) producer representatives to attend the regular meetings as guests. Many who did so were outspoken and helpful in the discussions, but naturally had no vote. The practice worked out satisfactorily, and was probably a reasonable compromise solution.

Timing

It was naturally difficult to keep the output of the subpanels and other assisting groups, such as the AMRA Evaluation Laboratory, abreast with the main panel. Standardized test procedures and analytical methods were needed from the very start, but these tasks generally took a few years to accomplish. The full effectiveness of the AMRA Laboratory was not achieved, because generally the selection from competing candidate alloys became mandatory before the AMRA tests could be completed. While the outputs of the subsidiary groups were occasionally "late", the products were reliable without exception, and no essential task was overlooked.

Time Schedule

Although an initial formal schedule had not been planned, this program has not adhered to the time schedule envisioned by its sponsors at the program initiation. Although most delays were the results of contractors' technical problems, some delays could have been avoided by increased emphasis on timing and by "pressure" from the Panel.

Concluding Remarks

This activity nominally has been a coordinated effort to achieve high quality refractory metal alloys in one product form, flat sheet. Because these same alloys are of interest for forging and tubing forms and because consolidation and ingot breakdown studied for sheet are prerequisites of all wrought forms, it can be said there has been considerable spin-off that has aided these other product forms.

The accomplishments were results of coordination among the military, the consumers, the fabricators, and the metal producers who became acquainted with each other's problems. Requirements were well publicized. The Services cooperated with each other to a high degree. Most important, the effort was focused. Only a few carefully chosen alloys were selected for development; only a limited number of the most important properties were measured, but in a way to permit needed comparisons, and a real effort was made to avoid unknown or unneeded duplication. Certainly the Government saved much money and time because of this selectivity and coordination. As a result there now exists a production base that can turn out a quality product. This was the prime objective of the program.

It would be difficult to say with conviction that the job is finished. The major objectives have been met but the Panel has recommended a modified Phase I and Phase II activity for several materials (see Table 4) that remains to be implemented. The Panel has recommended that specific responsibility be assigned for collection and dissemination of such information beyond the formal lifetime of the Panel.

Major benefits were derived by the focusing upon objectives, by narrowing the list of alloys for support, by getting people together to reveal and attach common problems. In an area such as high-strength

TABLE 4

Panel Recommendations in Current Program

Alloy	Phase I (Production)	Phase II (Prelim. Design Data)	Phase III (Fabrication)
<u>Fab. Mo</u>			
1/2ZrTi TZM	Universal Cyclops Universal Cyclops	Southern Research Southern Research	McDonnell McDonnell
<u>Unalloyed W</u>			
P.M. Arc Cast	Fansteel Universal Cyclops	Southern Research Not Recommended	Solar Super-Temp
<u>Tantalum</u>			
30Cb-7½V T-222 GE-473	Wah Chang Not Recommended Not Recommended	Not Recommended Recommended (a) Recommended (a)	----- Not Recommended Not Recommended
<u>Columbium</u>			
D-43 Cb-752 PS-85	du Pont Haynes (pilot) Recommended	Recommended (b) Recommended (b) Recommended (b)	Not Recommended Not Recommended Not Recommended
<u>High Strength Mo</u>			
Arc Cast TZC (c)	-----	-----	-----
Powder Met. TZM (c)	-----	-----	-----

TABLE 4 (continued)
Panel Recommendations in Current Program

Alloy	Phase I (Production)	Phase II (Prelim. Design Data)	Phase III (Fabrication)
Ductile $W^{(d)}$	-----	-----	-----
High Strength $W^{(d)}$	-----	-----	-----

Notes

- (a) Recommended Phase I production (not necessarily process development) of sufficient quantity of material to fix and define the production process and to provide material for Phase II evaluation of this pedigreed material.
- (b) Recommended collection and assembly of Phase II data (and Phase III where possible) of material already produced in Phase I.
- (c) Identified as candidate for preproduction program when requirements for high strength molybdenum warrant.
- (d) Alloys were identified that have exceptional promise. After additional laboratory optimization has been completed, at least a pilot study is recommended. (See Appendix II, page 120)

refractory metals, where the costs of the product and of development are high, the market small, and where the Government in the end is the major consumer, it seems imperative that the production industry and the consumer continue to get together in some working forum to provide mutual guidance.

The recently completed studies of the MAB Aerospace Applications Requirements Panel (Ref. 3) outlined requirements for all materials for propulsion systems (turbojet, turborocket, turboramjet, ramjet, liquid rocket, solid rocket, and electrical propulsion) and vehicle systems intended for operational capability in 1970. They reviewed devices, components, operational and environmental regime of components, and looked at fabrication requirements. For the propulsion systems alone they specified four sheet and plate requirements, three tubing requirements, three forging requirements, four coating requirements, and two thermionic device requirements for refractory metals. In reviewing fabrication requirements, it was found that 18 of 44 were due to the use of refractory metals. It was concluded that refractory metals will be a pacing item. The report broke down the problems of priority and came up with seven items on refractory metals in priority I. By identifying requirements so that orderly alloying and process development can proceed, it should be possible to avoid an expensive, inefficient crash program later.

In the Table, the Panel has not recommended further work in high-strength tungsten and molybdenum. This is because requirements were not specific enough to justify production development at this time. The AARP report suggests that such material will be a firm requirement soon.

Ductile tungsten alloys containing about 5 per cent rhenium recently have been reviewed by the Alloy Requirements and Selection Subpanel. It was recommended that additional laboratory optimization be conducted, and that a selected alloy or alloys be scaled up at least to the pilot level for demonstration of feasibility and determination of property data.

Some have proposed that whereas the Sheet Rolling Panel has concerned itself with bringing along process development of the required sheet, the Panel or future activity should concern itself with R&D in refractory metals as well. If a "working forum" concept for future activities in refractory metals can be developed, a forum where less time-consuming concern with contractor problems can be displayed, more attention to selected applied materials research and process development would seem proper and necessary.

The Panel clearly sees an immediate need for refractory metal tubing of the same alloys of columbium and tantalum endorsed in the sheet program. This coordinated activity should continue -- the preferred method is to continue the Tubing Subpanel.

Coatings of the refractory metals are the key to successful application of refractory metals in many propulsion and vehicle systems. The Coating Subpanel has established testing standards and is now evaluating specific coatings in several temperature-time spectrums. A coordinated approach in this area has been a major need for years. It is recommended that this activity also continue.

Specific recommendations are summarized at the beginning of this document.

TABLE 5

Publications of
The Refractory Metals Sheet Rolling Panel

<u>No.</u>	<u>Title</u>	<u>Date</u>
164-M (1-20)	Progress Reports of the RMSRP	
171-M	Joining of Refractory Sheet Metals	3/20/61
172-M	Report of Activities of Refractory Metals Sheet Rolling Panel, Superseded by 188-M	5/22/61
176-M	Evaluation Test Methods for Refractory Metal Sheet Materials, Superseded by 192-M	9/6/61
178-M	Report of the Subpanel on Analytical Techniques Refractory Metals Sheet Rolling Program	11/15/61
179-M	Report of the Subpanel on Consolidation and Fabrication, RMSRP	11/1/61
181-M	Report of Subpanel on Coatings, RMSRP	6/1/62
184-M	Report of Subpanel on Quality Specifications RMSRP	6/8/62
188-M	Status of Refractory Metals Sheet Rolling Panel	12/1/62
189-M	Evaluation Procedures for Screening Coated Refractory Metal Sheet	2/15/63
190-M	Quality Sampling Specification for Tungsten Plate	3/15/63
192-M	Evaluation Test Methods for Refractory Metal Sheet Materials, Superseded by 216-M	4/22/63
196-M	Recommended Quality Standards for Tungsten Sheet Produced in the Refractory Metals Sheet Rolling Program	1/22/64
201-M	Procedures for Evaluating Coated Refractory Metal Sheet, Supersedes MAB-189-M	8/10/64
208-M	Status of Refractory Alloy Tubing - 1964	8/16/65
210-M	Coating Technology - 1965, Oxidation-Resistant Coatings for Refractory Metals	11/65
212-M	Final Report - Refractory Metals Sheet Rolling Panel	3/66
216-M	Evaluation Test Methods for Refractory Metal Sheet Material, Supersedes 192-M	11/65

TABLE 5 (continued)

Publications of

The Refractory Metals Sheet Rolling Panel

<u>No.</u>	<u>Title</u>	<u>Date</u>
217-M	Cooperative Analysis Program on Refractory Metal Alloys	2/66
99-LM	Processing of Materials in INFAB	3/20/63
100-LM	Final Report of the Ad Hoc Infab Subpanel Refractory Metals Sheet Rolling Panel	3/26/65

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3. Report of Aircraft and Astronautics Applications Panel, Phase II, (Classified Secret), MAB-139-M(AA3), Materials Advisory Board, National Academy of Sciences-National Research Council, July 13, 1961 (Superseded by MAB-200-M, Vol. 2).
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6. Evaluation of Refractory Metals Sheet Rolling Panel Tungsten Sheet 9 July 1965, Contract NOW 63-0786d, Project 3478-62, Final Report September 1963-July 1965, Solar, a Division of International Harvester.
7. A Comparative Evaluation of the Formability of Tungsten Plate and Sheet by Spinning Techniques, July 15, 1965, Contract NOW 63-0542-c Final Report July 1963-July 1965, Super-Temp Corporation.
8. Report of Subpanel on Consolidation and Fabrication, MAB-179-M, December 1, 1961. Materials Advisory Board, National Academy of Sciences-National Research Council.
9. Joining of Refractory Sheet Metals, MAB-171-M, March 20, 1961, Materials Advisory Board, National Academy of Sciences-National Research Council.
10. Report of the Subpanel on Quality Specifications, MAB-184-M, June 8, 1962, Materials Advisory Board, National Academy of Sciences-National Research Council.
11. Quality Sampling Specification for Tungsten Plate, MAB-190-M, March 15, 1963, Materials Advisory Board, National Academy of Sciences-National Research Council.
12. Recommended Quality Standards for Tungsten Sheet Produced in the Refractory Metals Sheet Rolling Program, MAB-196-M, January 22, 1964, Materials Advisory Board, National Academy of Sciences-National Research Council.
13. Evaluation Test Methods for Refractory Metal Sheet Material, MAB-216-M, November 1965, Materials Advisory Board, National Academy of Sciences-National Research Council (Supersedes 192-M and 176-M).

14. Report of the Subpanel on Analytical Techniques, MAB-178-M, November 15, 1961, Materials Advisory Board, National Academy of Sciences-National Research Council.
15. Cooperative Analysis Program on Refractory Metal Alloys, MAB-217-M, February 1966, Materials Advisory Board, National Academy of Sciences-National Research Council.
16. Report of the Subpanel on Coating, Refractory Metals Sheet Rolling Panel, MAB-181-M, June 1, 1962, Materials Advisory Board, National Academy of Sciences-National Research Council.
17. Evaluation Procedures for Screening Coated Refractory Metal Sheet, MAB-189-M, February 15, 1963, Materials Advisory Board, National Academy of Sciences-National Research Council.
18. Coating Technology - 1965, Oxidation Resistant Coatings for Refractory Metals, MAB-210-M, November 1965, Materials Advisory Board, National Academy of Sciences-National Research Council.
19. Status of Refractory Metal Tubing - 1964, MAB-208-M, August 16, 1965, Materials Advisory Board, National Academy of Sciences-National Research Council.

SUBPANEL SUMMARIES

Previous pages were blank, therefore not filmed.

ROSTER

REFRACTORY METALS SHEET ROLLING PANEL

SUBPANEL ON ALLOY REQUIREMENTS & SELECTION

Chairman: Dr. Robert I. Jaffee
Senior Fellow
Department of Physics
Battelle Memorial Institute
505 King Avenue
Columbus, Ohio

Members

Mr. G. Mervin Ault, Associate Chief
Materials & Structure Division
Lewis Research Center, NASA
Mail Stop 105-1
21000 Brookpark Road
Cleveland, Ohio 44135

Mr. Alan V. Levy, Manager
Materials & Fabrication
Research & Development Department
Solid Rocket Operation
Aerojet General Corporation
Sacramento, California

Liaison Representatives

Mr. S. V. Arnold
Army Materials Research Agency
Watertown Arsenal
Watertown, Massachusetts

Mr. Irving Machlin
Materials Division, RRMA-23
Bureau of Naval Weapons
Department of the Navy
Washington, D. C.

Mr. I. Perlmutter, Chief
Physical Metallurgy Branch
Materials & Ceramics Division
Materials Laboratory
Wright-Patterson AFB, Ohio

Staff Metallurgist: Dr. Joseph R. Lane

Former Members:

Mr. L. Jahnke, General Electric
Mr. J. Maltz, NASA

Alloy Requirements & Selection Subpanel

Objectives

The duties assigned to the Alloy Requirements & Selection Subpanel were the following:

1. To determine the technical requirements for refractory metal sheet alloys in advanced defense and space systems,
2. To establish the state-of-the-art capabilities for refractory metal alloys,
3. To establish target properties which would be both achievable over the state of the art and also represent a sufficient advance to warrant the development effort within the program time frame,
4. To establish minimum development status to qualify a new alloy as a candidate for developmental support,
5. To review candidate alloys and recommend selected candidates for the sheet rolling program,
6. To follow changes in defense and space requirements and state-of-the-art alloy development and recommend modifications of and supplement to the sheet rolling program as needed.

Accomplishments

State of the Art

A meeting with producers of refractory metals at the outset of the program, December 1959, revealed that there were many more producers than the market warranted, alloy development was advanced only for molybdenum alloys, and strong production of large, thin-gage, aircraft-quality sheet of alloys was nonexistent. However, the energy and enthusiasm of the refractory metal producers, and their professed intentions to invest in major equipment to produce high-quality sheet products and alloy

development programs suggested to the ARS Subpanel that substantial improvements in the state of the art could reasonably be expected.

Requirements

Reviews of present and projected requirements for refractory metals in aerospace applications indicated that the projected requirements were much greater than present ones. The projected requirements depended upon authorizations of future aerospace systems, which were not firm. Present requirements were for systems that did not require large quantities of refractory metal sheet. The only requirement for refractory metal alloy sheet was in re-entry systems, where quality and fabricability were somewhat more important than strength at elevated temperature. A projected requirement for large tungsten sheets for liners in solid propellant rocket nozzles did not materialize because of technical difficulties. Thus, it was apparent that establishment of a capability for producing quality, high-strength, large-size, thin-gage sheet would be useful chiefly as a standby for future aerospace requirements.

Target Properties

Seven classes of refractory metal sheet were established: fabricable and high-strength classes for molybdenum, columbium, and tungsten, and a combined high-strength fabricable class for tantalum. Target properties were set up for each class and circulated to the producers and development laboratories. These served as a uniform basis for presentation of alloy candidates for consideration in the sheet rolling program. Perhaps more importantly, the refractory metal target properties provided guidelines for alloy development. The exchange of property information on candidates provided comparative information upon which alloys under development might be evaluated. The fact that the RMSRP alloy targets were substantially met by alloy candidates during the existence of the program may have been as much the result of the existence of the targets themselves as of the alloy development effort.

Recommendations of Alloys

The following alloys were recommended to be supported under the sheet rolling program:

Fab. Mo:	Mo-0.5Ti-0.8Zr(TZM)
Fab. Cb:	Cb-27Ta-10W-1Zr(FS-85)
Fab. Cb:	Cb-5Mo-5V-1Zr(B-66)*
Fab. Cb:	Cb-10W-1Zr-0.1C(D-43)**
Ta:	Ta-9.6W-2.4Hf-0.01C(T-222)
W:	See below

* Pilot-scale program

** Endorsement of Air Force support

In addition to the above recommendations, the RMSRP guided sheet rolling programs on powder metallurgy and arc-cast tungsten sheet, Cb-10W-2.5Zr (Cb-752), Cb-15W-5Mo-1Zr(F-48), and Cb-10Mo-10Ti(D-31). The Alloy Requirements and Selection Subpanel was not involved in recommending these compositions for the program. They endorsed the tungsten programs, but did not endorse F-48 or D-31.

A review of ductile tungsten alloys in July 1965, indicated that there were some alloys at the laboratory stage of development. These alloys were recommended for continued development in sheet form, at least to the pilot level.

A comparison of the Subpanel targets with the properties of the selected alloys as presented by the producer is given in Table 6. The dates listed under the alloy composition refer to the date of the presentation. Data on other candidates are given in the Appendix.

Recommendations to be Implemented (See Appendix)

Molybdenum

The Climax TZC alloy should be considered as a candidate for pre-production sheet rolling when the requirements for high-strength molybdenum

TABLE 6. Target Properties & Properties of Recommended Alloys (as presented)

Property	Molybdenum			Columbium				Tantalum	
	Fabricable	Target	Mo-0.5Ti-0.08Zr (TZM) May 20, 1960	Cb-27Ta-10W-1Zr (FS-85) Dec. 13, 1963	Cb-5Mo-5V-1Zr (B-66) Dec. 13, 1963	Cb-10W-1Zr-0.1C (D-43) Dec. 13, 1963	Fabricable	Target	Ta-10W-2.5Hf-0.01C (T-222) April 17, 1963
Room-Temperature Tensile									
Optimum	f_u - f_{ty} -10 ^(a)	f_u - f_{ty} -10	152-14-12 97-72-17	93-77-18 84-64-23	116-95-20 102-77-33	88-72-22 --	f_u - f_{ty} -15 ^(a) f_u - f_{ty} -15	-- 110-105-25	--
Recrystallized									
Elevated-Temperature Tensile									
Temperature, F	2000	2000	2000	2000	2000	2000	2400	2400	2400
f_u - f_{ty} -E1 ^(a)	75-60-(b)	50-40-(b)	74-65-5	46-44-22	52-43-32	47-45-12	35-28-(b)	53-38-20	53-38-20
Temperature, F	2400	2400	2400	2400	2400	2400	3000	3000	3000
f_u - f_{ty} -E1	50-35-(b)	20-15-(b)	48-40-8	24-21-61	26-26-87	27-25-25	25-16-(b)	25-24-24	25-24-24
Stress Rupture Strength, ksi									
Temperature, F	2000	2000	2000	30	29	33	2400	No data	No data
1 Hr	(b)	(b)	No data	25	--	29	(b)	--	--
10 Hr	(b)	(b)	"				(b)		
Temperature	2400	2400	"	16	11	15	3000	"	"
1 Hr	(b)	(b)	"	13	7	10	(b)	"	"
10 Hr	(b)	(b)	"				(b)		
Recrystallization Temperature, F	2600	2500	2500	2250	1900-2300	2400	State	No data	No data
50% in 1 Hr									
Transformation Temperature, F (4T)	-40	<RT	<RT	<-320	-100	-100(2T)	-320	<-320(2T)	<-320(2T)
Room-Temperature Bend Ductility									
Base	1 T	1 T	1 T	<1 T	<1 T	1 T	1 T	<2 T	<2 T
Weld	4 T	No data	No data	<1 T	3 T	<2 T	2 T	2.5 T (-250 F)	2.5 T (-250 F)

(a) Ultimate strength in ksi, 0.2% offset yield strength in ksi, percent elongation in 1 inch.

(b) To be furnished.

warrant. At the same time, Sylvania TZM alloy produced by powder metallurgy should be considered, since it has properties comparable to those of arc-cast TZC.

Columbium

The Fansteel FS-85 alloy (Cb-27Ta-10W-1Zr) and the duPont D-43 (Cb-10W-1Zr-0.1C) alloy were considered to be the two outstanding columbium alloy candidates, and both should be supported by production sheet rolling programs. Specifically, an evaluation-type sheet rolling program on FS-85 was recommended to the Navy (December 13, 1963) with sufficient material to be produced according to Fansteel's optimum schedule to the MAB quality specification for Phase I (Evaluation and Reproducibility Demonstration), Phase II (Design Criteria), and Phase III (Component Fabrication).

Tantalum

The GE-473 alloy (Ta-7W-3Re) and the Westinghouse T-222 alloy (Ta-10W-2.5Hf-0.01C) were identified as the two outstanding tantalum alloys so far developed. Because of no present or known future requirement for high-strength tantalum sheet, no recommendations for support were made. When and if such requirements appear, consideration should be given to sheet rolling programs on both alloys. (Note: The main panel recommended a "modest" sheet rolling program on T-222 and GE-473.)

Tungsten

Advanced tungsten alloys of both the high-strength and ductile classes have shown exceptional promise. After additional laboratory optimization has been completed, a selected tungsten alloy or alloys should be scaled up at least to the pilot level for demonstration of feasibility and determination of design data.

ROSTER

REFRACTORY METALS SHEET ROLLING PANEL

SUBPANEL ON ANALYSIS METHODS

Co-Chairmen: Professor L. L. Seigle
State University of New York
Department of Material Sciences
College of Engineering
Stony Brook, L. I., New York

Mr. Theodore D. McKinley
Research Supervisor
Pigments Department
E. I. duPont de Nemours & Co., Inc.
Experimental Station
Wilmington 98, Delaware

Members

Dr. James R. DeVoe, Chief
Radiochemical Analysis Section
Analytical Chemistry Division
National Bureau of Standards
Washington, D. C. 20234

Dr. Velmer A. Fassel
Institute for Atomic Research
Iowa State University
Ames, Iowa

Mr. William F. Harris
Research Department
Youngstown Sheet & Tube Company
Youngstown, Ohio

Dr. Manley W. Mallett
Battelle Memorial Institute
Columbus, Ohio
(now located at G. E.
St. Petersburg, Florida)

Liaison Representatives

AF/RTD Mr. C. D. Houston, Code MAYA, Wright-Patterson AFB, Ohio

Army Mr. Sam Vigo, Army Materials Research Agency, Watertown Arsenal
Watertown, Massachusetts

Navy Mr. I. Machlin, Bureau of Naval Weapons, Department of the Navy
Washington, D. C. 20360

BuMines Mr. R. A. Beall, Albany Metallurgy Research Center, Albany, Oregon

Staff Metallurgist: Dr. Joseph R. Lane

Former Member: B. F. Scribner, NBS

Analysis Methods Subpanel

Introduction

The task of the Analysis Methods Subpanel was to determine whether the precision and repeatability of chemical analysis of the refractory metals and alloys with existing techniques was satisfactory for the purposes of the Sheet Rolling Program, to ascertain the existence and nature of problems in analysis of these alloys, and to recommend courses of action to remedy such problems. As a first step, a review of cooperative analytical studies and government-sponsored research was conducted in 1961, as well as a survey of the experience of producers, users, and interested government agencies in analysis of refractory metals, in order to determine the state of the art, and identify problem areas in analysis. These studies, which are reported in detail in MAB-178-M, November 15, 1961, led to the following conclusions:

1. Analytical techniques for major alloying constituents in the refractory metals were probably adequate. However, this fact needed to be confirmed by interlaboratory cooperative programs on selected alloys. The main problem area in analysis was the determination of the interstitial elements oxygen, nitrogen, carbon, and hydrogen at low levels.
2. There was a great need for standard or reference samples of refractory metals and alloys to permit interested organizations to check their analytical methods.
3. Several interlaboratory comparison programs under way, or planned, were concerned with analytical techniques for unalloyed tungsten, tantalum, molybdenum, and columbium, but no similar activity for alloys existed. Therefore, the

Refractory Metals Sheet Rolling Panel should institute cooperative programs on specific alloys of interest to it, including analysis for both major alloying constituents and impurities.

4. An agency such as the Defense Metals Information Center, or the National Bureau of Standards, should be appointed as a clearing house for the collection and dissemination of information on the availability of standard samples, status of cooperative programs, validated methods of analysis, etc., for refractory metals.
5. Further government support was justified for research which might lead to new methods for determination of low levels of the interstitial elements in refractory metals and alloys.

Interlaboratory Comparison Program

A. Preparation of Reference Alloys

It was clear that an interlaboratory comparison program was essential in order to ascertain the precise degree of agreement which was being achieved among laboratories in analysis of refractory alloys, define more specifically the problems in analysis, and improve the levels of agreement. Therefore, a cooperative program was initiated in 1962. The National Bureau of Standards at that time was not in a position to furnish reference materials. Consequently, the Albany Metallurgy Research Center of the Bureau of Mines, under contract with the Bureau of Naval Weapons, was assigned in 1962 to prepare highly uniform samples of unalloyed W, T-111, TZM, and FS-85 to serve as reference materials representing alloys of the four major refractory metals. Details of the preparation and characterization of the reference alloys are given in the Albany Metallurgy Research Center Final

Report USBM-U-1100, February 10, 1964, as well as DMIC Report No. 220^{*}. Approximately 25 pounds of 1/4" diameter rod and 25 pounds of machined chips of W (rod only), T-111, TZM, and FS-85 were prepared and analyzed. The alloys are all of nominal composition with the residual interstitials at the lower end of the spectrum of levels occurring in practice.

B. Round Robin Testing

Early in 1964, the first round robin analysis of the reference materials was organized with 25 laboratories participating, including the analytical laboratories of all the organizations directly involved in the Refractory Metals Sheet Rolling Program. This round robin, completed in August 1964, confirmed an adequate degree of interlaboratory agreement in analysis for the major alloying constituents, with the possible exception of Zr in FS-85, and much poorer agreement in analysis for C, O, N, and H in most alloys. A second round robin for the interstitial elements was then decided upon with more closely specified procedures, and finally a third for oxygen along in FS-85 and TZM, the latter particularly to confirm the homogeneity of the reference materials. In addition, samples were issued to a number of laboratories for analysis of interstitials by mass spectrometry. Details of the procedures and results of these operations are reported in MAB Report 217-M and in DMIC Report 220^{*}. A summary of the results and conclusions from the round robins is presented in the following section^{**}.

C. Conclusions from Round Robin Testing

1. The level of agreement among most laboratories with existing procedures for major alloying constituents is adequate for practical purposes, except possibly for Zr in FS-85, where a coefficient of variation of 10% at the 1.0% level is rather high. (Part of this variation may be

^{*}"Comparison of Chemical Analysis of Refractory Alloys" by D. L. Chase

^{**}Mass spectrometer results were not yet available at the time of writing. These will be collected by D. L. Chase of DMIC.

due to inhomogeneity in the reference material. See Report MAB-217-M.). Average compositions, standard deviations and coefficients of variation are given below. Methods used include X-ray spectroscopic, emission spectroscopic, and wet chemical methods.

	<u>FS-85</u>			<u>TZM</u>		<u>T-111</u>	
	<u>Ta</u>	<u>W</u>	<u>Zr</u>	<u>Ti</u>	<u>Zr</u>	<u>Hf</u>	<u>W</u>
Average (%)	27.67	10.11	0.92	0.50	0.089	1.75	7.88
Std. Dev. (%)	0.67	0.22	0.092	0.013	0.0055	0.09	0.21
Coef. Var. (%)	2.4	2.2	10.0	2.6	6.2	5.1	2.7

2. With only little standardization of procedures, most laboratories can agree within reasonable limits in the determination of C and H in refractory metals, even at quite low levels, using existing techniques. A summary of the round robin data for the interstitials is given in the table below. The results for C and H fall on or near the C. V. goal line, meaning that for these elements at 100 ppm the achieved coefficient of variation among laboratories is 10%; at 10 ppm 20%; and at 1 ppm (for H) 40%. This level of agreement is considered to be satisfactory for almost all purposes.

	<u>Carbon</u>				<u>Oxygen</u>			
	<u>W</u>	<u>T-111</u>	<u>FS-85</u>	<u>TZM</u>	<u>W</u>	<u>T-111</u>	<u>FS-85</u>	<u>TZM</u>
Avg. (ppm)	9.1	17	10	230	7.3	14	68	7.1
Std. Dev. (ppm)	2.12	2.82	2.74	16	3.42	3.85	11.5	2.44
Coef. of Var. (%)	23.4	16.6	27.4	6.9	46.9	26.9	16.9	34.4

	<u>Nitrogen</u>				<u>Hydrogen</u>			
	<u>W</u>	<u>T-111</u>	<u>FS-85</u>	<u>TZM</u>	<u>W</u>	<u>T-111</u>	<u>FS-85</u>	<u>TZM</u>
Avg. (ppm)	6.3	18	43	16	0.47	24	1.3	0.49
Std. Dev. (ppm)	4.09	6.25	5.84	4.25	0.30	5.81	0.51	0.28
Coef. of Var. (%)	65.0	34.7	13.6	26.6	63.9	24.2	39.2	57.2

3. Interlaboratory agreement in the determination of low levels of O and N with existing techniques and equipment is much less satisfactory

than for C and H, but probably adequate for most practical purposes. At 10 ppm, the achieved coefficient of variation is about 40%, or twice the desired variation. The methods in use for the determination of O and N are inherently capable of the desired sensitivity. Reasons for lack of agreement reside in the details of the analytical procedures. At low levels of O, the surface oxide on specimens leads to high results. The amount of surface oxide present will vary widely with minor changes in surface preparation. A second likely cause for lack of agreement in O analysis is high and variable vacuum or inert gas fusion blanks in some laboratories. On the encouraging side, agreement between neutron activation and vacuum or inert gas fusion results for O is reasonably good, lending confidence in the basic accuracy of the fusion methods.

The method of sample dissolution is an important source of error in the determination of N. Excessive time required to take samples into solution leads to contamination from the atmosphere. Refractory nitrides, such as ZrN, which occur in many of these alloys, may be difficult to dissolve completely. Once the sample is in solution, the isolation and measurement of N can be easily and accurately accomplished.

Recommendations for Future Work

The principal recommendations for future work are concerned with analysis for O and N at low levels. While some discrepancies among laboratories might be resolved by a continued testing program entailing a close scrutiny of all steps in the analysis, more importance is attached to further research on analytical methods. Continued research to improve the precision of the vacuum and inert gas fusion methods for levels of oxygen and nitrogen below 20 ppm is recommended. Attention should be given to reducing the O content of the blank as well as to establishing optimum sample and bath sizes for precision at low levels. In particular, it would be desirable to explore the determination of O in low-oxygen materials by hard gamma irradiation which produces O^{15} . (Oxygen 15 decays with a half-life of 2.1 minutes which permits cleaning of the surface before counting, and thus allows

determination of the core oxygen without interference from adventitious surface oxide.)

Methods that assure the complete and rapid solution of bulk samples for nitrogen determination need to be developed and proven. Special attention must be given to the disposition of refractory nitrides in alloys and their influence upon the accuracy of chemical analysis.

Future Disposition of Reference Materials

One of the goals of the Subpanel was to prepare and have available for distribution a number of reference materials which could be used in the future by interested organizations to check their analysis methods. Remaining quantities of the four alloys used in the round robins have been turned over to the National Bureau of Standards for this purpose. NBS has agreed to announce the availability of these reference materials through its standard media and issue samples to qualified requesting laboratories, with a copy of DMIC Report No. 220. Data obtained by groups requesting samples will be reported to NBS and the Bureau will issue an annual report for a period of at least two years.

ROSTER

REFRACTORY METALS SHEET ROLLING PANEL

SUBPANEL ON COATING

Chairman: Mr. James J. Gangler
Materials Research Branch
National Aeronautics and
Space Administration
Washington, D. C. 20546

Members

Dr. C. A. Krier
Aero-Space Division, 88-03
The Boeing Company
P. O. Box 3707
Seattle, Washington 98124

Mr. Harry A. Pearl
R. D. 6, Landview Drive
Dix Hills
Huntington, Long Island
New York

Mr. Donald Kummer
Chief Ceramic Engineer
McDonnell Aircraft Corporation
Department 272
St. Louis, Missouri

Dr. David K. Priest, Director
Development & Engineering
Fansteel Metallurgical Corporation
Number One Tantalum Place
North Chicago, Illinois

Liaison Representatives

Lt. D. R. James
Code MAAE
Wright-Patterson Air Force Base
Dayton, Ohio

Mr. I. Machlin
Materials Division, Code RRMA-23
Bureau of Naval Weapons
Department of the Navy
Washington, D. C.

Mr. O. O. Srp
Code MAMP
Wright-Patterson AFB
Dayton, Ohio

Staff Metallurgist: Dr. Joseph R. Lane

Former Members:

M. Kushner, Boeing
A. V. Levy, Aerojet General
D. Cona, Boeing
R. I. Jaffee, Battelle
E. D. Sayre, G. E.

Coating Subpanel

Introduction

The Subpanel on Coating of the Refractory Metals Sheet Rolling Panel was established in 1960 for the purpose of reviewing and coordinating coating development programs and to make recommendations concerning coatings for the sheet alloys of molybdenum, columbium, tantalum, and tungsten, which are of interest to the Sheet Rolling Program. While the applications of refractory metals will involve many types of environments, the Subpanel decided to concentrate its efforts on oxidation resistant coatings for use in air atmospheres.

Early in the Subpanel's deliberations, it became apparent that coatings could not be discussed apart from the metal substrate. Coated refractory metal systems must be optimized and evaluated for specific applications. In this report a coated refractory metal system is defined as a composite material involving a coating of a particular composition on a specific metal substrate applied by a specified process.

The results of the Subpanel's initial review of the state of the art and its recommendations for coatings research development were tabulated, analyzed, and published in 1962 in Report MAB-181-M. In order to evaluate and recommend development and scale-up of specific coating systems for the sheet rolling program on the basis of significant and uniform property measurements, it was found necessary to formulate criteria for the evaluation of coated refractory metal. The Subpanel then proceeded to solicit and collate proprietary evaluation procedures from a dozen coating vendors and users. These procedures were also correlated with those developed by the Subpanel on Standardization of Test Methods of the Refractory Metals Sheet Rolling Panel, published in Report MAB-192-M. In 1963, the Subpanel on Coating published its "Evaluation Procedures for Screening Coated Refractory Metal Sheet" in Report MAB-189-M. Industrial acceptance and experience led to a refinement of these procedures and the development of

more advanced tests which appeared in 1964 in Report MAB-201-M, "Procedures for Evaluating Coated Refractory Metal Sheet". Although these test procedures were devised primarily to enable the Subpanel to evaluate and compare coating systems, they have also been accepted by many in the industry as standard tests, thereby tentatively satisfying an important need.

Sufficient time has not elapsed since the introduction of its test procedures and issuance of a final report to enable the Subpanel to make a comparative evaluation of all available coated refractory metal systems. However, because the Refractory Metals Sheet Rolling Panel is terminating its activities in 1965, the Coating Subpanel recently conducted its final review and evaluation of the state of the art of coating systems. The results of this review and recommendations for future coating activities are presented in Report MAB-210-M, "Coated Refractory Metal Technology - 1965".

The purpose of this summary report is to 1) briefly recount the Subpanel's activities, 2) summarize the state of the art, and 3) make recommendations for future coatings research and development.

State of the Art

It can now be concluded that a technology exists to coat columbium and molybdenum for short-time oxidizing applications where temperatures are as high as 2800° and 3000°F, and for substantially longer times at lower temperatures. Several promising developments are being pursued for tantalum and tungsten. Disilicides of the four refractory metals, applied by diffusion of silicon into the metal surface with such modifiers as Al, Cr, Ti, B, V, or the refractory metals themselves, currently represent the basis of most coatings. Aluminides offer the only practical alternative to the silicides, but are primarily used on columbium and tantalum alloys. The silicides are generally superior in performance to the aluminides, but the latter have been favored for fabricated hardware where slurry techniques have the advantages of simplicity and impose fewer restrictions on component design.

Aside from silicides and oxides, other refractory materials may be considered as candidate coating materials. Recent work has indicated that certain alloys of refractory metals possess sufficient oxidation resistance to be considered as candidate coating materials. Hafnium-tantalum alloys have been studied which show promising oxidation resistance at temperatures in excess of 3500°F.

In addition to the slurry techniques, pack cementation and the fluidized bed represent the most commonly used processes for production of coated refractory metal components. A review of the state of the art of this technology indicates that coating systems being used today can be best described as first generation. The full potential of available coating materials has not been achieved. A greater emphasis on processing should add great improvements in reliability, reproducibility, and end-use practicability. Slurry-diffusion techniques may meet these goals.

An important conclusion that cannot be over-emphasized is that a coating system must be selected and optimized for each alloy, component design, and the mission it is to perform. Optimized coated refractory metal systems will be produced only when there is a simultaneous integration of an alloy, component design, manufacturing sequence and coating system.

In order to minimize the expensive and destructive evaluation of coated hardware, a great need exists for non-destructive testing techniques. Current techniques are not adequate. Development of such inspection procedures is under way, but more effort must be undertaken.

In regard to the properties of coated refractory metals, the following conclusions reflect the state of the art:

1. Coated refractory metal will almost always exhibit a degradation in mechanical properties as compared to uncoated material when before-coating dimensions are used in the comparison.

2. A given coating composition and associated coating process can be modified to minimize the detrimental effect of the coating on the mechanical properties of a given alloy.
3. A given coating system can have appreciably different effects on the different refractory metals. No single coating chemistry or application process can be selected as the best available coating for all or even the majority of applications.
4. The oxidation protective life of coatings for refractory metals is significantly affected by alloy, temperature, pressure, temperature-time profile, strain and coating thickness. The effects of these influencing parameters are interdependent upon each other and all parameters will usually be present simultaneously in any given application of coated refractory metal.
5. The minimum coating life is considerably less than the average coating life at any given temperature if a statistically significant number of specimens is tested. The early coating failures are "defect failures" and the probability of a serious defect increases with increasing surface area and linear inches of edge (or increased number of specimens). Thus, the probable life of coated hardware is much less than the average life of coated coupons.
6. The use of current state-of-the-art coatings on refractory metal foils of less than ten mils thickness appears to be impractical.

The most significant shortcomings of the state-of-the-art coatings are:

1. Lack of reliable oxidation protection on hardware particularly at edges.
2. The increase in the ductile-to-brittle transition temperature due to coating.
3. Insufficient life at reduced pressure (below about 10 mm Hg) and high temperature (above 2500°F) in oxidizing atmospheres.

Finally, the review of the state of the art of coating technology has indicated that very few examinations have been made of diffusion effects, microstructure, and phase compositions and their relationship to mechanisms of coating protection or failure. The thermodynamics and kinetics of diffusion are closely related to coating life.

Each of the refractory metals systems has its own advantages and disadvantages. Conclusions pertinent to each follow:

Columbium

The foregoing statements apply equally to all the columbium alloys emphasized by the Refractory Metals Sheet Rolling Panel. There are more coated columbium alloy systems available today than there are for the other refractory metals. Coating chemistry is better understood for columbium and, therefore, more coating types are available. The favorable mechanical properties of columbium, such as low brittle-ductile transition temperature, weldability, and shop handling characteristics make columbium of greater interest to the industry. Columbium components requiring coating will tend to be larger and more complex than components of molybdenum due to the greater ductility and weldability of columbium. There are certain disadvantages, however.

Weldments (TIG) in high-strength columbium alloys such as D-42, FS-85, Cb-752 may be brittle at room temperature after coating.

Most columbium alloys, particularly the high-strength alloys, undergo an elongation and bend ductility minimum between about 800° and 1800°F. When coated, the elongation and bend ductility may be substantially reduced in this temperature range and within a very narrow temperature range these properties may be zero.

Molybdenum

Coated TZM and other molybdenum alloys have strong competition from the high-strength coated columbium alloys. Advantages for the coated molybdenum alloys are their greater resistance to creep, better fatigue life, and lower rate of failure propagation above 2700°F because no liquid oxide forms to slag the coating. Disadvantages of coated TZM are the delamination tendency, high ductile-to-brittle transition temperatures, notch and strain-rate sensitivity, fusion weld embrittlement, and embrittlement after recrystallization and grain growth. At the present time, the very low and unpredictable ductility of coated TZM at room temperature is the principal disadvantage in its use for components.

Tantalum

There are little data at this time for coated T-222 alloy. Coating technology for other tantalum alloys is in an early stage of development. Available test data for the behavior of coated alloys are incomplete, scarce, and often ambiguous. The best developed coatings are the modified Sn-Al and silicides. Their use is limited to temperatures up to approximately 3000°F in air at normal atmospheric pressure and to lower temperatures at reduced pressures. These temperatures are usually not high enough to utilize the desirable high temperature mechanical and thermal capabilities of tantalum alloys. No suitable high temperature diffusion barrier materials or concepts have been developed.

Modified Sn-Al coatings are presently favored over silicides for use in air at atmospheric pressure. They are generally resistant to oxidation at higher temperatures; possess better ductility and withstand greater plastic deformation.

Tungsten

The use of silicide coatings for the protection of tungsten is reasonably well established. Marginal improvements may be expected in this area, but coatings to be used at temperatures at which tungsten promises most usefulness (3500^o-4000^oF) cannot be clearly defined.

The use of oxide coatings appears to be the only reasonable choice for the protection of tungsten in oxidizing environments at temperatures in excess of 3500^oF.

Recommendations

1. Currently available aluminides and silicides coating systems for columbium and molybdenum should be further developed for greater applicability, and reproducibility, directed at high reliability particularly for hardware. The approach should emphasize improvement of process control, inspection methods, and elimination of defects particularly at edges.
2. Research should be conducted leading to new coating systems for temperatures up to 3000^oF, having high reliability on hardware. The approach should emphasize mechanism of coating failures and the role of coating composition and microstructure.
3. The slurry and gas plating processes should be emphasized over pack cementation and vacuum pack processes in new coating developments.
4. New coating concepts should be identified and encouraged for protecting tantalum and tungsten well beyond 3000^oF. Basic research is particularly needed here. For example, kinetics of diffusion in pure and mixed oxide systems need to be studied as well as in barrier materials, refractory alloys, and inter-metallic systems. Progress, particularly in coatings for tungsten and tantalum, is seriously being hampered because of a

lack of knowledge in this area. Additional information is also needed on mixed oxide-phase diagrams, free energies of all possible reactions in coating systems, mobility of anions and cations in oxides, the defect structure of oxides, low temperature oxidation of pest phenomenon, and the high-temperature interactions of coating materials and substrates. The capabilities and interests of the universities should be utilized in these basic research areas.

5. A broader program for developing non-destructive testing techniques should be undertaken. New techniques are required for detecting and determining the detrimental influence of cracks, compositional inhomogeneities, and variations in coating thickness.
6. The development of new or modified coating systems should give greater attention to attaining and stabilizing high emittance values.
7. Uniform screening and meaningful simulated environmental tests should be devised for the evaluation of coated systems for propulsion and power generation systems.
8. Improvements in the compatibility of coatings and braze alloys should be investigated.
9. Research should be conducted on the reasons for the increase of the brittle-ductile transition temperature of coated alloys and weldments over that of the uncoated alloys to find a method to avoid the embrittlement.
10. Research to improve the life of coating systems of reduced pressures and temperatures above 2500°F is needed.
11. Studies on the re-use and re-furbishment of coated refractory metal systems should be conducted. Such investigations should include techniques of inspection after one or more use cycles and coating repair.

12. The members of the Coating Subpanel recommend that the Materials Advisory Board establish a new panel on oxidation resistant coatings for refractory materials. The objectives of this panel would be to:

- (a) Define the current and future applications for coated high-temperature superalloys, refractory metal alloys, and graphite.
- (b) Define the performance requirements for these applications.
- (c) Formulate meaningful evaluation procedures with respect to the requirements to assist in the development of non-destructive tests to disclose defective coatings. This is a major current requirement.
- (d) Review the evaluation procedures of the current Coating Subpanel and either revise them to make them more meaningful and acceptable, or make a strong recommendation to the DOD that the established procedures be closely followed.
- (e) Continually review and advise on the technical value of funded R&D programs on coatings, and make pertinent recommendations regarding their future status.
- (f) Recommend new action by the DOD on the key technical problems of the state of the art.

ROSTER

REFRACTORY METALS SHEET ROLLING PANEL

SUBPANEL ON CONSOLIDATION AND PROCESSING

Chairman: Dr. William Rostoker
Metals Division
IIT Research Institute
Chicago, Illinois

Members

Dr. W. A. Backofen
Associate Professor of Metallurgy
Massachusetts Institute of Technology
Cambridge, Massachusetts

Mr. R. A. Beall
Project Coordinator
Melting Laboratory
U. S. Bureau of Mines, Region I
Albany, Oregon

Professor Morris E. Fine
Materials Research Center
The Technological Institute
Northwestern University
Evanston, Illinois

Dr. H. H. Hausner
Consulting Metallurgist
730 Fifth Avenue
New York, New York

Professor N. H. Polakowski
Professor of Mechanical Metallurgy
Illinois Institute of Technology
Chicago, Illinois

Staff Metallurgist: Dr. Joseph R. Lane

Consolidation and Processing Subpanel

Objectives

Many subpanels of the Refractory Metals Sheet Rolling Panel were formed because of their importance to some facet of the overall problem-- Analysis or Test Methods, for instance. Recognition that consolidation and processing were the heart of the problems involved in making high-quality sheet reproducibly, and that the topics were too broad to be handled by the main panel led to the establishment of this group. The Subpanel was formed in February, 1961, and disbanded after a final meeting in July 1961. The Subpanel was specifically asked to:

1. Examine existing procedures used in manufacture and identify problem areas resulting in poor quality and low recovery;
2. Explore possibilities for small and large modifications of existing practices which might lead to improvement in quality and recovery;
3. Recommend areas of research and development, which present judgment would indicate to be profitable, for support by the Department of Defense.

In addition to the six-man subpanel, numerous guests were invited to participate in the discussion. These visitors were selected because of their direct experience with the aspect being considered. The first meeting was devoted to consolidation, the second to hot working, and the third to cold working. In the discussion and the writing of the report, no attempt was made to provide a cookbook-type solution to the operations. Rather, the problems were approached broadly so as to determine the factors involved which affected quality, as the products are now being made and as they could be made under other circumstances.

For the consolidation phase, both the pressing and hot sintering of powder and the arc-casting route are routine commercial operations. The value of modified procedures such as sintering over 2500°C or the casting of slab-shaped ingots was discussed. Other conceivable methods of consolidation discussed are centrifugally cast thick-walled tubes, subsequently split and rolled, or shear formed to a thin-walled cylinder and then split and flattened.

Both furnace capability, as well as a desire to avoid oxidation and contamination, tend to restrict the temperature used for extrusion or forging. Problems in lubrication and with die materials are additional complicating factors. Nevertheless, higher temperatures may be needed, especially when limited machine capacity is coupled with newer alloys with high hot strength. The specific choices in hot breakdown are more closely related to economic factors than technical ones; processes such as forging which are appropriate for small orders are more apt to be chosen at this stage in commercial development.

Cold processing is employed when possible because of avoidance of contamination and because of the relative ease of obtaining dimensional control. In rolling, problems of gage control often reflect variations established during hot working. For maximum corrective action, 4-high cluster mills with front and back tension are indicated. Due to small orders and small-size sheets, relatively primitive hand mills are apt to be used. The report discusses the corrective grinding of sheet for gage control and contamination removal. The problems of laminations and texturing are also covered.

Assessment of Results

Following some broad recommendations (calling for government-supported research on lubrication and on deformation) eight specific projects are proposed, which could lead to improvements in quality or economics of refractory metal sheet.

Briefly, these projects are:

1. Evaluation of sintering tungsten billets above 2500^oC.
2. Exploration of feasibility of producing slab-shaped cast ingots.
3. Examination of alternate routes for tube starting stock.
4. Exploration of new lubricants, and of lubricity.
5. Development of extrusion die materials.
6. Pilot production of sheet on oilium equipment.
7. Exploration of corrective machining of strip.
8. Correlation of mill processing, texture, and mechanical properties.

In the three years since the report was issued, a number of programs have been contracted by the Services consonant with the recommendations in MAB-179M. These are itemized below.

1. Slab - Shaped Billets

Project No: 8-118

Contract No: AF33(657)-11294

Contractor: Oregon Metallurgical Corporation

OBJECTIVE: The objective of Phase III of this program is to develop a manufacturing process for casting Ta-30Cb-7.5V and TZC (Mo-1.25Ti-0.15Zr-0.15C) fine-grained sheet bar suitable for direct conversion into sheet. The 1" x 4" x 8" sheet bar will be centrifugally cast.

2. Slab - Shaped Billets

Project No: 8-204

Contract No: AF33(615)-1393

Contractor: Union Carbide Corp., Stellite Division

OBJECTIVE: The objective of this program is the development of a manufacturing process for converting Cb-752 cast slabs into thin-gage sheet (0.020") of uniform quality. The rolling

practice and slab quality control necessary to produce close tolerance, flat, Cb-752 sheet with reproducible mechanical and metallurgical properties will be defined.

3. Floturned Tungsten Sheet

Project No: 7-917
Contract No: AF33(600)-43034
Contractor: Wah Chang Corporation

OBJECTIVE: The objective of this program is to develop the floturning process to consistently produce large diameter tungsten thin-wall tubing for slitting into sheet. Sheet widths will be greater than 12". Diminishing requirements and technical difficulties in obtaining a large mandrel at elevated temperatures caused this program to be terminated.

4. Extrusion Die Development

Project No: 7-946
Contract No: AF33(657)-8798
Contractor: Nuclear Metals, Inc.

OBJECTIVE: (a) To develop a satisfactory die design for hot extruding of refractory metals embodying the use of a tool steel or similar base metal coated with a suitable material. (b) To develop a new die material such as an oxide or a carbide, suitably reinforced with a metallic fiber. To develop a die of a metallic compound with a performance measurable as in "a" and "b" above.

5. Extrusion Lubricants

Project No: 7-947
Contract No: AF33(657)-9141
Contractor: Thompson-Ramo-Wooldridge, Inc.

OBJECTIVE: The development of lubricants and application procedures to optimize the extrusion of structural shapes from aerospace materials over a broad range of extrusion temperatures.

6. Effect of Dilute Impurities on Tungsten

Project No: 6.21.44.01.1

Contractor: In-House, U. S. Army Materials Research Agency

OBJECTIVE: To determine how additives such as nickel exert a profound effect on the grain growth and sintering of tungsten and thus how to utilize this knowledge in producing optimum properties in tungsten. To determine the volume diffusion and grain boundary diffusion constants in tungsten.

7. Zone Refining of Cemented Tungsten-Base Alloys

Project No: ORD TB5-002

Contract No: DA-11-022-505-ORD-3092

Contractor: IIT Research Institute

8. A Comparative Evaluation of the Formability of Tungsten Plate and Sheet by Spinning Techniques

Contract No: NOW 63-0542-c

Contractor: Super-Temp Corporation

OBJECTIVE: Tungsten plate from three sources are to be evaluated for spinability and shear formability.

9. Fabrication of Wide Tungsten Sheet by Point Deformation Techniques

Contract No: NOW 61-1046c

Contractor: Wah Chang Corp.

OBJECTIVE: Evaluate shear spinning techniques for producing wide tungsten sheet.

10. Influence of Heat Treatment on Physical Metallurgy

Project No: 735101-3M

Contract No: AF33(657)-11742

Contractor: Lockheed Missiles and Space Company

OBJECTIVE: To establish the physical metallurgical effects of processing wrought refractory metal products. The interactions of alloy composition and thermal and mechanical history are to be related to the structures of finished products. Emphasis is on the effects of variations in the latter stages of processing.

11. Influence of Processing on Physical Metallurgy

Project No: 735101-3F

Contract No: AF33(657)-11231

Contractor: IIT Research Institute

OBJECTIVE: To establish a rational basis for the processing of refractory metal wrought products by applying the principles of physical metallurgy. The relationship of composition and process history to structure and properties of finished products is to be determined. In contrast to other effort, emphasis is on effects of variations in earlier stages of processing and evaluation (extrusion, forging, sheet bar).

12. Development of Refractory Metal Foil

Project No: 651-G

Contract No: AF33(657)-9384

Contractor: Metals and Controls, Inc.

OBJECTIVE: To explore the problems of the production of reproducible high quality refractory metal foil, and to develop the optimum processing parameters. Included was development of required evaluation procedures and establishment of final quality specifications.

13. Development of Wide Refractory Metal Foil Process

Project No: 7-987

Contract No: AF33(657)-8912

Contractor: E. I. duPont de Nemours and Co., Inc.

OBJECTIVE: To develop a manufacturing process for the production of high quality refractory alloy foil in 100-foot coils up to 24 inches wide and in thicknesses down to 0.001 inch.

ROSTER

REFRACTORY METALS SHEET ROLLING PANEL

AD HOC INFAB SUBPANEL

Chairman: Mr. David Goldberg, Manager
Structural Materials & Processes
Astronuclear Laboratory
Westinghouse Electric Corporation
Pittsburgh, Pennsylvania 15236

Members

Dr. Robert I. Jaffee, Senior Fellow
Department of Physics
Battelle Memorial Institute
Columbus, Ohio

Dr. James Wong
National Research Corporation
Cambridge
Massachusetts

Liaison Representatives

Mr. George Glenn
Code MATB
Wright-Patterson Air Force Base
Dayton, Ohio
Alternates: Mr. V. dePierre
Captain D. Iden

Mr. I. Machlin
Materials Division, RRMA-23
Bureau of Naval Weapons
Department of the Navy
Washington, D. C.

Staff Metallurgist: Dr. Joseph R. Lane

Former Member: Mr. L. P. Jahnke, G. E.

Ad Hoc Infab Subpanel

The series of processing variables which were examined for the production of molybdenum alloy sheet included ingot breakdown and rolling in Infab. This installation (the name derived from fabrication in inert atmosphere), located on the property of Universal Cyclops Steel Corporation, had been separately funded by the Navy.

Although operations in Infab had been carried on since 1961, uncertainty existed as to the actual inertness of the atmosphere and the metal processing functions which the facility might best perform.

Since Infab processing had been selected as an element in molybdenum alloy sheet production under the DOD sheet rolling program, a brief investigation was activated by the Refractory Metals Sheet Rolling Panel. An ad hoc committee was formed (without formal approval, and comprised entirely of members of the main panel), as shown on the roster. Quoting from the first report (MAB-99-LM) of the Subpanel, the objective was:

"The Refractory Metals Sheet Rolling Panel has authorized the organization of a working party to accumulate the existing information relevant to the achievement of the objectives of Infab. Its mandate is to review the data for its validity and significance, to draw such conclusions as may be justified, to make specific recommendations for further work necessary for competent judgment, and to explore by discussion the nature of other functions which the Infab facility might perform in the spectrum of manufacturing activities associated with refractory metals."

The Subpanel solicited data from Universal Cyclops and the few other organizations thought to possess relevant information.

As a result of the discussions, a program to measure the contamination incurred during Infab processing was recommended. The Air Force responded by making alterations in their existing contract with Cyclops to encompass the desired experiments.

It was recognized that two considerations were involved. First was the effective inertness of the atmosphere. The second, broader problem, came down to the possible economic advantages of avoiding contamination compared with conventional processing followed by scalping, for example.

Two reports were issued. The first was a letter report (99-LM) dated March 20, 1963. It was observed in this report that suitable contamination data are meager, but that existing evidence was sufficient to conclude that surface contamination was not precluded. To provide a basis for judgment as to alloys or operations for which Infab might be advantageous, a program for measuring the kinetics of contamination was detailed.

The final report (100-LM) was issued March 26, 1965. Answers to three questions were sought:

1. What are the unique technical and possible economic advantages of Infab?
2. What additional research and development may be needed to establish the value of the facility?
3. What processes are uniquely suited to Infab (turbine forging, extrusion, welding, forge ultra-high strength alloys, large weldments, etc.)?

By this time, and as a result of recommended measurements made under the Air Force contract, sufficient data were at hand to state that the original target of working metals without contamination or degradation of properties had been met. While the atmosphere was unreactive, iron contamination from the rolls did result.

On the second point, additional research which might be needed, the Subpanel pointed to a complete lack of a firm basis for defining any economic advantage for Infab.

Two conclusions were reached relative to the third question, processes uniquely suited for Infab. On the one hand, no metalworking processes were identified as being uniquely appropriate for inert gas processing. On the other hand, there is a presumption that continuing development of high strength alloys may result at some future time in alloys which will require true hot working under conditions which may best be handled in an Infab-type installation.

The continuing uncertainty regarding Infab concerns economic aspects. At this point, no one knows if savings will result from commercial processing in Infab or in a similar type of installation. At present, alternative routes to Infab processing are employed with reasonable success, including: canning, pickling of contaminated surfaces, warm or cold processing, and locally installed gas chambers. However, Infab, because of its large size, flexibility, and good atmospheric control may offer advantages in the future.

It would seem to be appropriate for the obtaining of such data to be left to those who can utilize and profit from it now that the technical feasibility has been demonstrated.

ROSTER

REFRACTORY METALS SHEET ROLLING PANEL

SUBPANEL ON JOINING

Chairman: Dr. William Rostoker
Metals Division
IIT Research Institute
Chicago, Illinois

Members

Mr. Alan F. Lusto
Technical Administrator
Fansteel Metallurgical Corp.
North Chicago, Illinois

Mr. William N. Platte
Metallurgy Department
Westinghouse Electric Corp.
Research Laboratories
Pittsburgh, Pennsylvania

Mr. Harry Schwartzbart
Assistant Director
Metals & Ceramic Research
IIT Research Institute
Chicago, Illinois

Staff Metallurgist: Dr. Joseph R. Lane

Joining Subpanel

The objective of the Joining Subpanel was to make an assessment of the state of the art to enable recommendations for needed research to be drawn up. The report of the Subpanel (MAB-171-M) is dated March, 1961, after which the Subpanel disbanded. For this reason the comments below may no longer hold.

A number of visits were made by Subpanel members to capitalize on available knowledge. A first conclusion reached was that this body of information was very fragmentary.

Discussion and Results

Joining by riveting, brazing, and welding was considered. Welding received the greatest attention, and the situation regarding each of the four metals is discussed in the report in turn. The advantages and disadvantages of gas, electron beam, resistance, and ultrasonic welding processes are covered.

Most knowledge was available (at the time of writing) for molybdenum alloys. There were problems with contamination (and hence brittleness), especially with sheet made from powder, but the real gap was knowledge concerning the structural performance of weldments. One aspect of this problem is the notable strain-rate sensitivity of molybdenum. The big problem of welding molybdenum by any process (assuming excessive grain size in the nugget is avoided) involves the heat-affected zone in which cracks are easily produced during welding. EB welding is notable for the small size of heat affected zone which results, but very accurate fit-up is required.

Some columbium alloys are readily weldable, but others tend to be brittle either as-welded or after heat treatment. The few tantalum alloys known at the time were weldable, and most alloys developed subsequently are also weldable. In a structural sense, tungsten and molybdenum are

unweldable. The problems of tungsten welding are partly masked by the brittleness of the base metal. TIG and EB tungsten welds may be made successfully, but porosity problems with sheet made from powder exist in the same manner as with molybdenum.

The report concluded that development was going on actively in many industrial organizations, with the trend toward the use of automatic equipment. EB welding has apparent advantages, which may be a function of the voltage used. Test criteria are needed to permit an evaluation of brazed joints. All four refractory metals can be fusion welded successfully if an appropriate technique is used. Spot welds also appear possible, but a background of experience is largely lacking. Brazing is generally limited to temperatures below which most hardware would operate. Riveting is feasible, but may involve weight, cost, or reliability (in coated assemblies) problems.

Conclusions and Recommendations

Four recommendations were offered:

1. A program to define tests for simulation of the various severities of service is needed.
2. Development of brazes with high remelt temperatures should be pushed.
3. A research program leading to a better understanding of solid state joining is needed.
4. Alloy development should always include consideration of the great importance of weldability.

This subpanel report was the first which became available in the sheet rolling operation. In the intervening four years, substantial progress has been made, particularly by the fabricators of hardware. Probably most effort has been on columbium alloys. While it would be presumptuous to say that all problems have been solved, it does seem clear

that use of refractory metals will not be inhibited by the joining problem -- a fear which led to the formation of the subpanel. Such achievements as the McDonnell ASSET vehicle and the Douglas Nike tungsten blast tube attest to the advanced state of the art. Problems are no longer with merely making a joint, but with keeping the transition temperature low and the joint efficiency high by maintaining the usual controls over porosity, recrystallized metal, etc.

A detailed analysis of the aerospace joining requirement for the 1965-1980 time period and the deficiencies in our present capability is contained in "Report No. 2 of the Aerospace Manufacturing Techniques Panel," MAB-139-M(AMT-2), dated October 15, 1963.

ROSTER

REFRACTORY METALS SHEET ROLLING PANEL

PHASE II SUBPANEL

Chairman: Mr. G. M. Ault, Associate Chief
Materials & Structure Division
Lewis Research Center, NASA
MS 105-1
21000 Brookpark Road
Cleveland, Ohio 44135

Members

Dr. Robert Jaffee
Senior Fellow
Department of Physics
Battelle Memorial Institute
505 King Avenue
Columbus, Ohio

Mr. Alan V. Levy, Manager
Materials & Fabrication
Research & Development Dept.
Solid Rocket Operation
Aerojet General Corporation
Sacramento, California

Mr. L. P. Jalunke, Manager
Materials Development Laboratory
Advanced Engine & Technology Dept.
General Electric Company
Cincinnati, Ohio 45215

Mr. John T. Stacy
Senior Group Engineer
The Boeing Company
Aero-Space Division
P. O. Box 3707
Seattle 24, Washington

Liaison Representatives

AF/RTD Mr. I. Perlmutter, Code MAMP, Chief, Physical Metallurgy Branch
Materials & Ceramics Division, Materials Laboratory, Wright-
Patterson AFB, Ohio

Mr. George Glenn, Code MATB, Wright-Patterson AFB, Ohio

Navy Mr. J. Maltz, Materials Division, Bureau of Naval Weapons
Department of the Navy, Washington, D. C. (presently with NASA)

Staff Metallurgist: Dr. Joseph R. Lane

Phase II Subpanel

Objective and Approach

The objective of the Phase II activity was to obtain needed mechanical and physical property design data from the "pedigreed" sheet produced in Phase I. The determination as to which specific tests were to be run and at what temperatures and times (for long-time data) was the primary interest of this Subpanel. The decision was made that the minimum data should be obtained that, in the judgment of the designers, would permit them to evaluate the general characteristics of the materials as to their potential for use in advanced structures. It was decided that all the design data that ultimately may be sought by a designer should not be obtained at this point in the program because the specific applications for the product were not known and because the fabrication experience gained in Phase III would influence the future use of the material.

The procedure used to develop the list of properties that would meet the above criterion follows:

1. The Aerospace Industries Association (AIA) was contacted for its advice. AIA proposed that we use the report ARTC-12 "Basic Properties for Comparative Evaluation of Structural Metallic Materials" (revised July 1, 1960) as a basis, and that we determine most (but not all) of the Priority 1 and 2 data from Charts I and II of that report.
2. The Phase II Subpanel analyzed this proposal of AIA, added its own more detailed knowledge of the scope and nature of the sheet rolling program, and from this study, produced a modified list.

3. This list was submitted to AIA. The response was a hearty endorsement of the proposed program and its details. Minor constructive comments were received from certain individual AIA representatives and most were incorporated. This final program is included herein as Table 7 and has been used for the formal Phase II programs.

Status and Recommendations

The Navy established a Phase II program at Southern Research and two of the products (TZM, and unalloyed W) of Phase I of the sheet rolling program are being evaluated as prescribed (see Table 2, main panel report). The documentation of the important properties of the pedigreed sheet has been of considerable use. It is rare that an opportunity is provided for the determination of mechanical and physical properties that the designers need on materials for which the production development and method has been documented. A continuation of this approach is recommended.

The choice of materials for inclusion in the Phase II evaluation has been the responsibility of the main panel. They have recommended that, in addition to the Phase II programs now under way, Phase II programs be conducted for T222, GE473, and FS85, and that Phase II data being determined randomly by a number of laboratories for Cb752 and D43 from previous Phase I AF programs be collected and assembled in a single document. (See Table 4 main panel report.) To date the follow-up of these recommendations has been initiated for only FS85 (by the Navy). These recommendations should be implemented.

TABLE 7

Basic Required Properties for Comparative Evaluation of Structural Metallic Materials for Phase II

Section	Item	Properties	Matl. cond.	Expo- sure time	Properties required at indicated temperatures Of (All tests longitudinal unless otherwise indicated)										
					H.T. ¹	1200	2000	2200	2500	2800	3000	3500	4000	4500	
MECHANICAL PROPERTIES															
I Tension	A. Stress-strain curves ³ 1. To at least 0.3% offset; with complete curve at as many temps as possible		Optimum and re- crys. and coated (18)	1/2 hr	All ⁴	All	All		All		All	Ta, W	Ta, W	Ta, W	
	B. Tensile properties 1. Ultimate, yield (0.2% offset) and elongation ⁵ and C. Modulus of elasticity		Optimum and re- crys.	1/2 hr											
	II Compression A. Stress-strain curves ³ to at least 0.3% offset B. Compressive yield (0.2 offset) and C. Comp. modulus		Optimum	1/2 hr	All ⁴										
III Tensile notch sensitivity ⁷				1/2 hr	Determine from diagrams of IIA										
IV Shear ultimate strength	Optimum and re- crys.		Optimum and re- crys.	1/2 hr	All										
	VI Bearing strength A. Ultimate (e/D of 1.5) B. Yield (2% strain) (e/D of 1.5)		Optimum	1/2 hr	All										
	VIII Creep in tension B. Creep strengths 2. 1.0% total defor- mation ¹¹ to 100 hrs		Optimum	1/2 hr	All										
IX Thermal Stability ¹² A. Under load	Optimum		Optimum												
	Bend data Bend test (3t at 10"/min; at temp. increments to 50° F above transition temp.; longitudinal and transverse.)		Optimum, recryst, and coated (18)	All											
	Minimum bend radius (Specify span and ram rate)		Optimum recryst.	All											
Weld data Fusion weld joint ef- ficiency (19)	As welded and after heat treat if desired		As welded and after heat treat if desired	All											
	Spot weld joint efficiency in shear		As welded and after heat treat if desired	All											
	PHYSICAL PROPERTIES														

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ROSTER

REFRACTORY METALS SHEET ROLLING PANEL

PHASE III SUBPANEL

Chairman: Mr. Roger Perkins
Staff Scientist
Materials Sciences Laboratory
Lockheed Miss'les & Space Company
Palo Alto, California

Members

Mr. Donald A. Honebrink
Research Engineer
Structures Technology Department
Aero-Space Division
The Boeing Company
Seattle 24, Washington

Mr. Howard Siegel
Senior Technical Specialist
Material & Process Department
McDonnell Aircraft Corporation
St. Louis, Missouri

Mr. Edward D. Weisert
Principal Scientist
Department 591-355
Rocketdyne Division
North American Aviation
Canoga Park, California

Dr. James Wong
National Research Corporation
Cambridge, Massachusetts

Staff Metallurgist: Dr. Joseph R. Lane

Phase III Subpanel

Objectives and Approach

The objective of Phase III programs was to evaluate the quality and fabricability of sheet produced in Phase I. A secondary objective was to delineate optimum procedures for unit fabrication operations and the general limits of fabricability for Phase I sheet. Activities of the Subpanel were concerned with definition of the basic needs of the Department of Defense and industry with respect to information on the fabricability of "pedigreed" refractory metal sheet alloys. Recommendations were made to the Contracting Agency for Phase III programs to assist in planning and guiding the technical efforts.

The first task of the Subpanel was to define a general scope for technical efforts that would apply to all Phase III programs. It was recommended that five major elements be incorporated in each program:

1. Part Selection: Select for fabrication studies small parts or subassemblies typical of current or anticipated use. Manufacturing sequences should include a broad variety of unit fabrication processes.
2. Limits of Formability: Determine the capabilities with respect to fabrication of Phase I sheet and the limits within which good quality parts can be made for each unit process.
3. Metallurgical Investigation: Evaluate the general structure and quality of sheet and correlate with the response to forming.
4. Forming Tests: Fabricate by repetitive means a sufficient number of parts to demonstrate consistency (or lack of) in response to forming by optimum procedures.

5. Structural Testing: Conduct mechanical or structural tests to determine the properties or performance capabilities of fabricated parts and subassemblies.

Detailed recommendations for the scope of technical effort in each of these five areas were made to serve as a guide in planning future Phase III research activities.

Two programs had been funded before the Phase III Subpanel was created. The Subpanel, therefore, concerned itself with evaluating the existing programs in terms of the objectives and scope for such programs as defined above. Where necessary, changes in direction of technical effort were recommended to align these programs with desired Phase III activity. A third program funded after the organization of the Subpanel was planned in accordance with the basic recommendations of the Refractory Metals Sheet Rolling Panel.

During the course of the three fabrication programs, the Subpanel met periodically with representatives of the contracting agency and the contractors to review progress and future plans. These reviews were used to accelerate the dissemination of technical information, both from the contractors to the Panel and from the Panel to program monitors. The primary purpose, however, was to guide the Subpanel in making sound and useful recommendations for future work.

Summary of Program Activities and Results

1. Mo-Base Alloy Sheet: One program on the fabrication of Mo-alloy sheet was conducted by McDonnell Aircraft Corporation, St. Louis, (Navy-BuWeps Contract No. NOW-64-0456-c). The main contributions of the Subpanel were recommendations to concentrate efforts on: (1) evaluation of pedigree TZM sheet produced in Phase I and to minimize work on Mo-0.5Ti sheet; and (2) fabrication of small parts and subassemblies typical of utilization

in ASSET heat shield panels rather than a large fin and rudder assembly as planned; and (3) evaluation of specific unit forming processes and determination of forming limits and basic sheet quality. These recommendations were implemented by the contracting agency with a consequent delay in the program. The technical results, however, more than justified the changes that were made and delays involved.

Results of this study indicated that the Phase I TZM sheet was of excellent quality and had a somewhat greater latitude and flexibility with respect to forming than current commercial production material. Minimum forming temperatures were appreciably lower than those currently in use for TZM sheet. Reproducibility in repetitive forming of parts was good. Problems with edge laminations still were found, however, in shearing, punching, or blanking operations. Preliminary results indicate good structural properties in fabricated parts and subassemblies.

2. Tungsten Sheet and Plate: Two programs were conducted on the forming of Phase I tungsten sheet produced from powder metallurgy billets: (1) a shear spinning evaluation of plate conducted by Super-Temp Corporation, Santa Fe Springs, California; and (2) a forming evaluation of sheet conducted by Solar, Division of International Harvester, San Diego, California. Both programs were contracted by Navy BuWeps.

Activities of the Subpanel with respect to the spinning program were concerned with: (1) assistance in preliminary evaluation of sheet structure and history on ability to spin and form parts; (2) a recommendation to conduct a program on evaluation of spinning quality; and (3) recommendations for minor technical modifications during the course of the work. Preliminary studies were conducted through the courtesy of Aerojet-General Corporation, Sacramento, California, at the request of the Subpanel to demonstrate the feasibility of forming sheet produced by the Phase I program at Fansteel. As a result of this work, basic product mixes of Phase I sheet for use in forming studies were delineated and recommendations made to conduct forming evaluation programs.

The program on spinning could not be oriented completely with the general objectives and scope of Phase III programs due to prior firm contractual commitments. Results of the investigation were very useful, however, and contributed greatly to knowledge in fabrication of tungsten parts by shear spinning. It was found that spinning characteristics of a given part were more sensitive to small changes in forming parameters than to large differences in the structure and prior history of tungsten plate. The Phase I sheet was of excellent spinning quality and had exceptionally good mechanical properties in fabricated forms. These results were exceptionally gratifying on the basis of prior judgment by producers and users of tungsten sheet that indicated the history of manufacture of Phase I sheet was not conducive to good spinning characteristics.

The forming evaluation program conducted by Solar was planned in accordance with subpanel recommendations. Panel activities therefore were concerned with technical liaison and recommendations for selection of final components for structural evaluation. Applications of direct interest to Navy BuWebs were reviewed in making the final recommendation for manufacture of a supersonic ram jet combustor. Results of this program clearly defined forming limits for a wide variety of unit operations and demonstrated the good quality and formability of Phase I sheet.

3. General Remarks: The information and data obtained as presented in the final reports on these programs will have broad utility in the years ahead. The results of each program conducted are of far more value to the Department of Defense and industry since they comprise a complete and detailed evaluation of fabrication capabilities for specific areas of application of a given material. This approach is believed to have been more effective than the alternate approach of doling out samples of sheet materials to various fabricators and attempting to collect, correlate, and disseminate the bits and pieces of data obtained. Invariably, the latter approach leaves many gaps in knowledge of material capabilities and often provides data that cannot be correlated due to lack of standardized evaluation procedures.

Recommendations

With respect to future fabrication evaluation programs of Phase I sheet alloys, it is recommended that Phase III programs be considered for Cb and Ta alloys going through pilot scale sheet production programs. These could be combined with Phase II programs which have been recommended for these alloys to provide an evaluation of mechanical properties, general quality, and fabrication capabilities in one report. The past Phase III programs, in effect, tended to duplicate some of the tests conducted in Phase II studies. Efforts in these two regions could easily be coupled by a slight reduction in scope of activities in each area. Information on mechanical properties and fabricability similar to that obtained for TZM and W will be needed for the tantalum and columbium-base alloys. The approach used by the Phase III Subpanel was effective in assisting the contracting agency in planning and conducting the evaluation programs, and demonstrated how harmonious working relations between an advisory subpanel, the contracting agency, and contractors could be developed and used to plan and direct a well coordinated program. It is recommended that similar approaches be tried in future activities like those of the RMSRP.

ROSTER

REFRACTORY METALS SHEET ROLLING PANEL

SUBPANEL ON QUALITY SPECIFICATIONS

Chairman: Mr. John T. Stacy
Senior Group Engineer
Aero-Space Division
The Boeing Company
P. O. Box 3707
Seattle 24, Washington

Members

Mr. S. E. Bramer
Douglas Aircraft Corporation
Santa Monica, California

Mr. Robert Freeman
Marketing Manager
Refractory Metal Division
Climax Molybdenum Co. of Mich., Inc.
Ann Arbor, Michigan

Mr. Basil T. Lanphier
Chief Metallurgist
Carpenter Steel Company
Reading, Pennsylvania

Staff Metallurgist: Dr. Joseph R. Lane

Former Members: Mr. Arnold Rustay
Wyman-Gordon

Mr. George Timmons
Climax Molybdenum

Quality Specifications Subpanel

Objective

The primary objective of the Quality Specifications Subpanel was to assure the production of refractory metal sheet in the Phase I effort under controlled conditions in order to establish the desired uniformity and quality of sheet available for subsequent evaluation in Phases II, III, and IV. While a desirable objective for any type of material usage, such control was highly desirable in the case of refractory metal alloys. For example, refractory alloy data which were available at the start of Phase I indicated that refractory metal and alloy sheet was plagued with the following deficiencies: inconsistent properties within a sheet and from sheet-to-sheet, poor surface quality, insufficient flatness, tendency to delaminate, high and variable ductile-brittle transition temperatures and inconsistent recrystallization temperatures.

Operations

In order to assure the quality desired in refractory metal and alloy sheet, it was necessary to establish minimum criteria for the acceptance of such sheet from the DOD contractors. Sheet which met these criteria would be accepted for subsequent evaluation on other phases of the refractory metal sheet rolling program. Such sheet would be known as "pedigreed sheet". To insure compliance, controlling specifications were required.

Initially, an attempt was made to assemble enough existing data on the then most available refractory alloy sheet, Mo-0.5w/o Ti, to permit the establishment of property levels by statistical methods. When it became apparent that the data were insufficient to be handled statistically, various alloy producers were contacted to determine what minimum property and quality levels could be met with their alloys and their production methods. Based on the information received, a specification was drafted to delineate minimum acceptable properties and quality for a variety of

refractory metals and alloys. A review of the draft by the main panel indicated that such a specification would neither establish the desired uniformity of properties nor assemble sufficient data to permit subsequent analyses in order to set minimum standards. Hence, it appeared desirable to prepare a specification along the lines of a test plan, outlining requirements for sampling and testing. Based on this philosophy, a quality sampling specification was prepared.

Results

The first quality sampling specification was issued June 8, 1962, as Report MAB-184-II, "Report on the Subpanel on Quality Specifications, Refractory Metals Sheet Rolling Panel". The specification covered columbium, molybdenum, tantalum, tungsten and their alloys. Types of tests and methods of sampling were specified. Since the emphasis was on quality, explicit levels of properties were purposely largely omitted, permissible variations about a mean value or within a range being designated. Coverage included identification of sheet and lot; a sampling plan; visual, dimensional, and sonic inspection; tensile, bend transition temperature, metallographic, hardness, and recrystallization tests; and a detailed format for reporting the test results.

A second quality sampling specification was issued for tungsten plate: MAB-190-M, dated March 15, 1963, "Quality Sampling Specification for Tungsten Plate". This specification was similar to that above, but added requirements for penetrant inspection, grain size, grain shape, and specified minimum reduction in area. These added requirements appeared mandatory to assure shear-spinning quality.

The third specification issued differed from the previous two in that it set up required standards and it applied only to tungsten sheet (MAB-196-M, dated January 22, 1964, "Recommended Quality Standards for Tungsten Sheet Produced in the Refractory Metals Sheet Rolling Program"). In essence, this specification was the fruition of the sampling plan spelled

out in the first specification (MAB-184-M) wherein the data obtained on Navy Contract NOW-60-0621-c by the Fansteel Metallurgical Corporation were used to establish tensile minimums, thickness tolerances, flatness tolerances, and an elevated temperature bend test requirement for various thicknesses of tungsten sheet.

Assessment

In assessing the results of the work of the Quality Specifications Subpanel, several items are of particular interest. First, the guidance established by the issuance of the general quality sampling specification (MAB-184-M) was of value in detailing for the DOD those items of quality which are of importance to the users of refractory metal sheet. Likewise, the imposition of this documented requirement on DOD contractors educated the refractory metal sheet industry and promoted the production of quality sheet. The use of a sampling specification to control quality and, at the same time, to accumulate data for standardization such as was done in the case of tungsten sheet has been useful in elucidating the quality and mechanical properties of production sheet. These data resulted then in a firmly based quality standard or specification. However, to date, the only firmly based specification to be prepared on the DOD programs has been that for powder metallurgy tungsten sheet (MAB-196-M).

Data of the type required by the quality sampling specification, MAB-184-M, are available for the columbium alloy, D-43 (Cb-10W-1Zr-0.1C). Some data on a pilot production contract involving FS-85 (Cb-27Ta-10W-1Zr), have recently become available. Also, similar data are available for the Cb-752 alloy (Cb-10W-2.5Zr). Based on these data specifications such as that written for tungsten sheet (MAB-196-M) can be prepared. In the case of tantalum alloys, no data are available to permit the preparation of corresponding specifications. However, such a specification can be prepared for molybdenum alloys, either Mo-0.5Ti or TZM.

In summary, it will be noted that the initial mode of operation, that of preparing specifications for property and quality levels from existing alloy data, was not effective and that it was necessary to obtain data according to a test plan type specification to generate the required information. These data were immediately applicable to the preparation of realistic specifications. Hence, in any future operations of this type, it is recommended that a quality sampling specification similar to MAB-184-M be prepared and instituted as part of the contractual requirements in alloy development contracts involving some production of alloy. This second mode of operation was very successful; however, it should have been instituted at an earlier date in the life of the Panel to have realized its full benefits.

ROSTER

REFRACTORY METALS SHEET ROLLING PANEL

SUBPANEL ON STANDARDIZATION OF TEST METHODS

Chairman: Mr. L. M. Raring, Manager
Research & Development
Fansteel Metallurgical Corp.
Number One Tantalum Place
North Chicago, Illinois

Members

Mr. Donald A. Douglas, Jr.
Supervisor, Metallurgy Division
Oak Ridge National Laboratory
Oak Ridge, Tennessee

Mr. Michael J. Manjoine
Astronuclear Laboratories
Westinghouse Electric Corp.
P. O. Box 10864
Pittsburgh, Pennsylvania

Mr. Roger Perkins
Staff Scientist
Materials Sciences Lab.
Lockheed Missiles & Space Co.
3251 Hanover Street
Palo Alto, California

Staff Metallurgist: Dr. Joseph R. Lane

Standardization of Test Methods Subpanel

The objective of this Subpanel was to provide a standard basis for the test evaluation of refractory alloy sheet produced under various Department of Defense development programs. Established ASTM and other standards did not include sufficient definition of methods for the oxidation protection of refractory metals during test and of temperature control requirements at the higher test temperatures.

The Subpanel membership was selected from research and development engineering organizations actively engaged in the application of refractory metal sheet. A number of producers and users of refractory metal sheet were first canvassed by the Subpanel to determine their practices and to solicit comments with regard to refractory metal sheet evaluation. As could be expected, the results showed a wide divergence in detailed practices, most of which was attributed to differences in equipment or in design applications. As an example of the latter, laboratories concerned principally with high-temperature aerospace structures were mostly interested in short-time properties. Other groups needed longer time test data for their design purposes. The producers' requirements were usually different than either of these. Routine quality control testing was generally designed to reflect important manufacturing effects. At a meeting on January 31, 1961, in Cleveland, Ohio, the results of the survey were analyzed and discussed. A tentative set of standards was drawn up defining basic test conditions but, at the same time, providing for minor variations in procedures to meet specific equipment, design, and material needs. The test methods were also coordinated with properties criteria as established by the Alloy Requirements and Selection Subpanel. Following this first meeting, comments were again solicited from a number of interested laboratories and after these had been evaluated, a report was published on September 6, 1961 (MAB-176-M). This report received considerable comment by both users and producers of refractory metal sheet. A number of questions were raised concerning specific practices related to tensile strain rate, bend test practices, and temperature measurement. The Subpanel met

again on June 25, 1962, in Washington to review these comments. After detailed consideration and communications with various laboratories and producers, a final draft was agreed upon and a revised report, MAB-192-M, was issued April 22, 1963. Several additional tests of interest to fabrication evaluation and preliminary design analyses were also included in the second edition.

Additional comments were received following release of MAB-192-M. After review by the Subpanel, a revised draft was submitted to the main panel on September 16, 1965. It was decided to publish the revised draft as the third edition of the report on "Evaluation Test Methods for Refractory Metal Sheet Material". This report, MAB-216-M, will outline standard methods for the following tests and determinations:

Tensile	Weld Evaluation
Compression	Recrystallization Temperature
Shear	Fatigue
Bend	Thermal Conductivity
Bearing	Thermal Expansion
Creep-Rupture	Specific Heat
Delamination	Modulus of Elasticity

These procedures form a "standard" basis for comparison of refractory sheet metal by different laboratories. By definition, "standard" methods cannot define all details needed to meet special design or fabrication requirements. Such "special" tests must be designed to specific environmental, strain, and temperature conditions as dictated by the design application.

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REFRACTORY METALS SHEET ROLLING PANEL

STEERING COMMITTEE

Chairman: Mr. G. Mervin Ault
Associate Chief
Materials & Structure Division
Lewis Research Center, NASA
MS 105-1
21000 Brookpark Road
Cleveland, Ohio 44135

Member

Dr. Robert I. Jaffee
Senior Fellow
Department of Physics
Battelle Memorial Institute
505 King Avenue
Columbus, Ohio

Liaison Representatives

Mr. George Glenn
Code MATB
Wright-Patterson Air Force Base
Dayton, Ohio

Mr. I. Machlin
Materials Division, RRMA-23
Bureau of Naval Weapons
Department of the Navy
Washington, D. C.

Staff Metallurgist: Dr. Joseph R. Lane

Former Member:

L. P. Jahnke, G. E.

Steering Committee

The diversity of problems before the Panel led to the formation in April 1962 of a Steering Committee. The purpose of the group was to review these problems and recommend items to be brought forward for decision by the main Panel. In effect, the Steering Committee established the agenda for meetings of the main Panel. Principal decisions were the assignments to be made to subpanels, and the contractors selected to present a resume of progress at panel meetings. A selection of guests appropriate to each meeting was also made.

As the Panel operation came near an end, the remaining tasks became quite evident. Meetings of the Steering Committee became brief, and the last meeting was held in January 1965.

On the subjects discussed at the Steering Committee meetings, none reappeared more often than that of the desired coverage of the Panel: should other mill forms, particularly tubing, be included? An outgrowth of this debate was an early informal group that reviewed the tubing problem (reported in Progress Report Numbers 3 and 6), and the subsequent establishment in April 1964 of a formal Tubing Subpanel.

Another typical decision related to coverage in terms of materials. New developments in chromium alloys were reviewed several times (always with the same conclusion: maintain awareness of research within Steering Committee but exclude from Panel consideration.).

The final problems related to possible changes in mode of operation, and to formulation of an orderly plan to disband the refractory metal sheet rolling activity.

ROSTER

REFRACTORY METALS SHEET ROLLING PANEL

SUBPANEL ON TUBING

Chairman: Mr. William Thurber
Development Department
Parma Research Laboratory
Union Carbide Corporation
Parma, Ohio

Members

Mr. David C. Goldberg, Manager
Structural Materials & Processes
Astronuclear Laboratory
Westinghouse Electric Corporation
P. O. Box 10364
Pittsburgh, Pennsylvania 15236

Mr. L. M. Raring, Manager
Research & Development
Fansteel Metallurgical Corporation
Number One Tantalum Place
North Chicago, Illinois

Dr. Robert I. Jaffee
Senior Fellow
Department of Physics
Battelle Memorial Institute
Columbus, Ohio

Mr. John T. Stacy
Senior Group Engineer
The Boeing Company
Aero-Space Division
Seattle, Washington 98124

Liaison Representatives

AF/RTD Mr. George Glenn, Code MATB, Wright-Patterson AFB, Ohio

Army: Mr. S. V. Arnold, Army Materials Research Agency, Watertown Arsenal, Watertown 72, Massachusetts

NASA: Mr. J. Maltz, Materials Research Program, Code RRM, National Aeronautics & Space Administration, Washington, D. C. 20546

Mr. Thomas Moss, Lewis Research Center, NASA, 21000 Brookpark Road, Cleveland, Ohio 44135

AEC: Mr. Andrew Van Echo, Reactor Development Division, Atomic Energy Commission, Washington, D. C. 20545
Formerly Mr. S. S. Christopher

Staff Metallurgist: Dr. Joseph R. Lane

Tubing Subpanel

Goals of the Subpanel

An increasing attention to refractory metal tubing triggered by anticipated aerospace and nuclear applications, coupled with the fact that most of the tubing alloys were identical to those being developed for sheet, led to the formation of a Tubing Subpanel in March 1964. An ad hoc group had made a preliminary report in mid-1961 indicating no immediate requirement for a tubing activity. Subsequently, an informal coordination group involving NASA, AEC, and the Air Force was instituted, largely to maintain continuity in the production, test, and evaluation phases. After it became apparent that many of the problems just solved for sheet were pertinent to tubing (for example, alloy selection and test methods), the desirability of a Tubing Subpanel was indicated.

The following charter was established for the Tubing Subpanel:

- I. Assess requirements for refractory metal tubing
- II. Define state of the art
 - A. Production capability for individual alloys
 1. Size range
 2. Quality
 3. Experience
 - B. Equipment and process status
 - C. Quality control and test methods status
 - D. Tubing properties
- III. Establish technical objectives for development of tubing technology
 - A. Process development needs
 - B. Target tubing properties for various alloy bases
- IV. Follow current programs, recommend new programs, and indicate distribution plans and uses for tubing produced on Government-funded programs.

In fulfilling this commitment, several commercial tube mills were visited and technical briefings were presented to the Subpanel by tubing manufacturers. Both conventional and novel fabrication processes were inspected. These visits supplemented the composite knowledge already available to the Subpanel. Culmination of this phase of the Subpanel's activities was the publication of Report MAB-208-M, "Status of Refractory Alloy Tubing - 1964". The report covers, in large measure, Items I, II-A, B, C, and III-A cited above in the charter.

Typical requirements defined for refractory alloy tubing and described in detail in MAB-208-M include nuclear reactor fuel element cladding, piping for space power systems, (e.g., Rankine and Brayton cycle, Thermionic) liners of regeneratively cooled rocket nozzles, structural members in aerospace vehicles, liners for barrels of small-bore weapons, and internal components for nuclear propulsion rockets. Limited industrial applications, particularly in various highly corrosive media, also exist. Definition of requirements and establishment of the production state of the art led to the following specific recommendations for further process development in the refractory metal tubing field:

1. The flow-turning process under development by TMCA appears to be attractive as a means for producing refractory metal tubing from either extruded or welded tube rounds. Practicability of this method for producing tubing of the more ductile refractory alloys should be evaluated.
2. Both new and conventional methods of tube manufacturing should be extended to higher temperatures. For example, drawing and tube reducing of tubing at temperatures between 500-1500°F should be developed in order to capitalize on alloys which cannot be worked near room temperature.
3. An exploratory study to define fabrication problems associated with special shapes such as tapered tubes should be undertaken.

4. Improved methods for producing composite tubes should be developed, particularly composites comprised of a conventional alloy and a refractory alloy.
5. Investigation of novel tube producing processes should be maintained to establish feasibility and permit comparisons of product quality and operating economy with conventional methods.
6. Studies to better understand deformation processes for making tubing are recommended, including development of tests whereby fabricability of new alloys into tubing may be determined.

Some of these recommendations are currently being implemented, while others have not yet been considered in detail for support. The Subpanel strongly urges that all of these proposals receive careful consideration for inclusion in future Government-funded developmental programs.

Target Properties for Tubing

Another major function of the Subpanel was to establish target properties for refractory metal alloys which could be fashioned into tubing with properties superior to those currently available. In large measure, the Tubing Subpanel was able to capitalize on the extensive ground work laid by the Alloy Requirements and Selection Subpanel of the RMSRP in establishing similar target properties for refractory metal sheet. However, in specifying target properties for tubing, greater emphasis was placed upon creep and stress-rupture characteristics. This reflects a principal need for refractory metal tubing in power applications and space propulsion missions of long duration. The target properties established for refractory metal tubing are cited in Table 2. These target properties are considered to be achievable combinations of strength and ductility beyond the present state of the art for refractory metal tubing alloys. It is recognized, of course, that not all advanced systems will demand such strength properties. Present alloy capabilities are, in fact, adequate for many contemplated

TABLE 8. Targets for Refractory Metal Tubing Alloys

	Cb	Ta	Mo	W
<u>Room Temperature Tensile</u>				
UTS	*	*	*	*
YS	*	*	*	*
Elongation, %	15	15	10	6
<u>Elevated Temperature Tensile</u> **				
2000 F	50-40 *	--	75-60 *	--
2400 F	30-25 *	50-40 *	60-50 *	--
3000 F	--	35-25 *	25-15 *	40-30 *
3500 F	--	15-10 *	--	25-15 *
<u>Creep Strength</u>				
<u>2200 F</u>				
1% - 100 hr	12	24	15	--
1% - 1000 hr	8	16	10	--
Rupture - 100 hr	20	40	25	--
<u>2400 F</u>				
1% - 100 hr	7	20	9	--
1% - 1000 hr	5	12	6.5	--
Rupture - 100 hr	*	*	*	--
<u>3000 F</u>				
1% - 100 hr	--	5	4	7.5
1% - 1000 hr	--	3	2.5	4.5
Rupture - 100 hr	--	10	8	15
50% Recrystallization Temp. 1 hr	2600	3000	3200	3600
<u>Transition Temperature, F</u>				
4T Bend	-100	-100	RT	RT
<u>Room Temp. Bend Ductility, T</u>				
Base	1T	1T	4T	4T
Weld	2T	2T	State	State

* To be furnished.

** Lists in order UTS, 0.2% YS, and % Elongation.

State the following:

Fabrication experience	Lamination tendency
Density	Notch sensitivity
Thermal expansion coefficient	Flarability
Emissivity	Response to flattening
Modulus of elasticity	Resistance to alkali metal
Oxidation and contamination	corrosion
characteristics	Nuclear cross section
Coating experience	

systems. However, it is a matter of historical record that designers are always quick to exploit superior material properties either through reduced weight or improved design margins.

The diverse applications for refractory alloy tubing often require specific properties or combinations of properties other than mechanical. For example, in Rankine cycle systems resistance to attack at elevated temperatures by alkali metals dictates inclusion of getter elements, such as zirconium or hafnium, in refractory alloy compositions. In nuclear fuel cladding, even more complex chemical restraints are imposed. Further, the strength requirement is different for each of these two applications. Tubing for fuel cladding must have high strength, while tubing for Rankine system piping requires good weldability and fabricability, strength being less important. In either case, ductility under biaxial stress should be appreciable (~5%). Thus, when tubing is used in complex engineering structures, strength considerations may become secondary.

One further comment on Table 8 is in order: Since sheet is generally available prior to tubing, several of the specified properties are referenced to sheet rather than to tubular mill products. Such property tests are useful screening tools prior to committing an alloy to tubing development program.

Need for Coordinating Group

In comparing these briefly summarized accomplishments with the goals of the Subpanel as defined by the charter, it appears that at this writing only Item IV requires major pursuit. There was unanimous agreement among the Subpanel members that to perform this function a formal interagency coordinating group for refractory metal tubing should exist. Since it was concluded that available refractory alloys are, in general, suitable for the contemplated applications, a major alloy selection or alloy development activity is not required. Rather, the focus should be on improving fabrication capabilities for refractory metal tubing.

The proposed group would operate as the focal point for information exchange and discuss, coordinate, and recommend Government-funded programs. It is realized that specific applications involve unique problems, but in each instance there should be sufficient relevant technology to make profitable a coordinated enterprise.

Major attention would be directed to the process metallurgy of refractory metals. However, this need not exclude cognizance of closely related programs on reactive metals where processing similarities exist. Agencies currently having active or planned programs in these areas include the Air Force, Army, AEC, and NASA and, thus, wide interservice interest is anticipated.

In making this recommendation for a follow-on tubing activity, the Subpanel recognizes the existence of an Ad Hoc AEC group on tungsten tubing and possible interagency groups concerned with the metallurgy of vanadium and chromium. These in no way diminish the strength of the recommendation. Although the tubing activity could be carried out by an informal committee without sponsorship, the Subpanel feels that operation under MAB aegis would be much more satisfactory. With MAB sponsorship, domination of the activity by any one agency would be avoided, participation by guests from industry would be practical, prestige (and presumably therefore functional capability) of the group would be enhanced, and formal channels for information dissemination would be available.

APPENDIX I

LETTER OF ASSIGNMENT

OFFICE OF THE DIRECTOR OF DEFENSE RESEARCH AND ENGINEERING

Washington 25, D. C.

June 18, 1959

Dear Dr. Bronk:

The Bureau of Aeronautics has initiated a Refractory Metals Sheet Rolling Program, expansion of which is expected both with Bureau of Aeronautics funds and expected supplemental funds from DOD.

Because of the importance and complexity of the program and the many diversified interests in it, the Bureau of Aeronautics has requested the assistance of the Materials Advisory Board in the form of an Advisory Committee, to function in a manner similar to that of the advisory group to the Bureau of Aeronautics for the Titanium Sheet Program.

It is requested that the above committee be established after consultation with the Bureau of Aeronautics as to details. It is understood that this office will be kept advised of the progress of the work under this assignment.

It is understood that this assignment is acceptable to the National Academy of Sciences - National Research Council, and will not require funds beyond the current contract appropriations.

Sincerely yours,

J. R. Townsend
Special Assistant

Dr. Detlev W. Bronk
President
National Academy of Sciences
2101 Constitution Avenue, N. W.
Washington, D. C.

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APPENDIX II

ALLOY REQUIREMENTS & SELECTION SUBPANEL

Chronology of Meetings

Listed below is a chronology of the meetings held by the Alloy Requirements & Selection Subpanel, and the purposes for which they were held:

- (1) November 3, 1959: Chicago, Illinois
To establish ground rules for consideration of candidates and set a schedule for activities of the Subpanel.
- (2) November 20-December 2, 1959: Washington, D. C.
To meet with producers of refractory metals to orient them about the Refractory Metals Sheet Rolling Program and to determine the status of refractory metal development.
- (3-a) January 20, 1960: Sacramento, California
Discussion of refractory metal requirements in solid rockets with Aerojet General.
- (3-b) January 21, 1960: Los Angeles, California
Alloy Requirements & Selection Subpanel meeting to set preliminary target properties.
- (3-c) January 22, 1960: Los Angeles, California
Meeting with ARTC Group to discuss aerospace requirements.
- (4) February 19 and 29, 1960: Washington, D. C.
Establishment of mechanical-property targets for refractory metal sheet alloys.
- (5-a) May 17, 1960: North Chicago, Illinois
Presentation of molybdenum candidate by Fansteel Metallurgical Corporation and Marquardt Aviation Corporation.
- (5-b) May 18, 1960: Detroit, Michigan
Presentation of molybdenum candidate by Climax Molybdenum.
- (5-c) May 19, 1960: Cincinnati, Ohio
Presentation of molybdenum candidate by General Electric, Evandale.
- (5-d) May 20, 1960: Bridgeville, Pennsylvania
Presentation of molybdenum candidate by Universal-Cyclops Steel Corporation.
- (5-e) May 20, 1960: Pittsburgh, Pennsylvania
Alloy Requirements & Selection Subpanel executive session to discuss molybdenum alloy recommendations.

- (6) July 6-7, 1960: Washington, D. C.
Presentation of columbium alloy candidates by producers.
- (7) August 24, 1960: Washington, D. C.
Setting procedures for uniform evaluation of refractory metal alloy candidates.
- (8) January 24-25, 1961: Columbus, Ohio
Producer presentations on columbium alloy candidates.
- (9) March 1, 1961: St. Louis, Missouri
Alloy Requirements & Selection Subpanel executive session to recommend columbium alloys.
- (10-a) March 22, 1961: Los Angeles, California
Discussion of refractory metal requirements with Space Technology Laboratory.
- (10-b) March 23, 1961: Sacramento, California
Discussion of refractory metal requirements at Aerojet General.
- (10-c) March 24, 1961: Livermore, California
Discussion of refractory metal nuclear requirements with Lawrence Radiation Laboratory.
- (10-d) March 29, 1961: Tremonton, Utah
Discussion of solid rocket requirements with Thiokol Chemical.
- (11) August 8, 1961: Washington, D. C.
Alloy Requirements & Selection Subpanel executive session to discuss columbium alloy recommendations.
- (12) November 2-3, 1961: Washington, D. C.
Producer presentations on tantalum-base alloys.
- (13) February 20, 1962: New York, New York
Alloy Requirements & Selection Subpanel executive session to set future plans.
- (14) March 26, 1962: Washington, D. C.
Additional columbium producer presentations and consideration of columbium sheet alloys.
- (15) April 17-18, 1962: Alexandria, Virginia
Alloy Requirements & Selection Subpanel executive session on recommendation of columbium sheet alloys.
- (16) May 14, 1962: Cleveland, Ohio
NASA requirements discussion, and review of AAAP refractory sheet metal requirements.

- (17) November 30, 1962: Washington, D. C.

Producer presentations on high-strength molybdenum and tungsten alloys.

- (18) April 17, 1963: Washington, D. C.

Producer presentations on high-strength molybdenum, tungsten, and tantalum alloys.

- (19-a) June 25, 1963: Oslo, Norway

- (19-b) September 4, 1963: St. Louis, Missouri

Discussion of refractory metal requirements and ARTC discussion of response to questionnaire.

- (20) December 10-13, 1963: Los Angeles, California

Discussion of columbium alloy recommendations, and additional presentations by Westinghouse, Fansteel, and Wah Chang.

- (21) May 21, 1964: Washington, D. C.

Producer presentations on tantalum and tungsten alloys.

- (22) July 13-14, 1965: Washington, D. C.

Producer presentations of ductile and high-strength tungsten alloys.

Status of Refractory Metal Development - 1959

The December 1959 presentations by the producers of refractory metal products afford a good starting point from which to project the activities of the Alloy Requirements & Selection Subpanel. Presented below is a summary of the alloys and ingot and sheet status as indicated by producer presentations:

Molybdenum

(1) Climax Molybdenum Company of Michigan was producing alloys from 8" diameter ingots, which were extruded and converted to sheet by various subcontractors. Their alloys were Mo-0.5Ti, Mo-0.5Ti-0.07Zr, and Mo-0.05Zr.

(2) Sylvania was producing sheet molybdenum alloys by sintering of isostatic-pressed billets, 5-1/2" x 11" x 24" long, in hydrogen furnaces, and rolling. They produced unalloyed molybdenum and had an Mo-1TiO₂ alloy under development.

(3) General Electric, Evendale, in collaboration with Universal Cyclops,

was producing pilot arc-cast ingots, weighing 100 pounds, 8 inches in diameter, of Mo-0.5Zr and TZC alloy, which were extruded and rolled.

(4) Fansteel was producing powder-metallurgy molybdenum, starting with ingots 6 inches in diameter maximum. In alloys, they were developing powder-metallurgy Mo-0.5Ti.

(5) Westinghouse (Bloomfield Works) was producing 2-inch-diameter powder-metallurgy billets of proprietary dispersed second-phase molybdenum alloys. They also produced an Mo-0.1C alloy, developed for Army Ordnance for improved hot-gas erosion and transverse strength.

(6) Universal Cyclops was concentrating on process development, but had produced production-size ingots of Mo-0.5Ti-0.07Zr, Mo-0.5Ti, and Mo-0.5Zr. These ingots were arc cast 8 inches in diameter, extruded, and rolled.

Columbium

(1) General Electric, Evendale, was well along with process development on F-48, made by arc casting, extrusion, and rolling. Also, they had processed a more oxidation-resistant, lower-strength alloy (F-50) containing titanium. Compositions were not disclosed at the time. Ingots were 6 inches in diameter, weighing 125 pounds.

(2) Union Carbide, in collaboration with other companies such as Allegheny Ludlum, Crucible, Universal Cyclops, and Westinghouse, was engaged in an active alloy-development program centered around a series of proprietary alloys. The alloy development level was based on 4-inch-diameter ingots.

(3) Du Pont was midway in setting themselves up as an integrated columbium producer and had a series of titanium-containing alloys, D-31 (Cb-10Ti-10Mo) and proprietary D-41 and D-42 under investigation at the 3-inch-diameter-, 50-pound-ingot level. They also had produced a D-31, 8-inch-diameter, 280-pound ingot. Du Pont was collaborating with Thompson Products Company (now Thompson-Ramo-Wooldridge) on secondary fabrication of columbium alloys into hardware. They were also planning their Baltimore metal-conversion facility to be installed by 1960.

(4) Temescal was concentrating on electron-beam-melted columbium- and tantalum-base alloys. Their columbium-alloy candidates contained 15 to 20 per cent tantalum and 15 to 20 per cent tungsten. Their tantalum alloys were based on additions of about 10 per cent tungsten. Their largest ingot was 5 inches in diameter. The alloys were hammer forged and then rolled to strip.

(5) Fansteel was working with arc-cast columbium alloys at the 4-inch-diameter ingot level of the FS-80(0.7Zr-0.050), FS-82(33Ta-0.7Zr-0.050), and FS-83, a proprietary alloy which later evolved as FS-85.

(6) Allison Division of General Motors, from a 1956-58 Air Force study, evolved a columbium alloy designated GMR-Nb-1085 of undisclosed composition. Its properties were similar to those of the General Electric F-50 composition.

(7) Wah Chang had a joint columbium-alloy-development program with Boeing, and was producing ingots 4 inches in diameter by a combination of electron-beam and arc-melting methods. No information was disclosed on their alloy-development efforts, but they had considerable production experience in producing F-48 ingots for General Electric.

(8) Westinghouse Research Laboratory had a columbium-alloy development program at the 150-gram-button level with typical alloy composition like Cb-5Mo-5V and Cb-5Mo-5V-1Zr (later turned out to be B-66).

Tantalum

(1) Fansteel indicated that they had been producing Ta-7.5W (under the name of Tantalloy) by powder-metallurgy methods for electron-tube application for over 30 years, and were now procuring electron beam melted ingots of Ta-10W from Temescal for processing into sheet. They had not yet rolled any Ta-10W, but were aiming at a maximum 24-inch wide, 72-inch long sheet size.

(2) Temescal pioneered electron-beam melting of tantalum, and were successful in producing 5-inch-diameter Ta-10W ingots, which they were having rolled to 6- to 12-inch wide sheet.

(3) Westinghouse Research Laboratory had a Government alloy-development contract, and were producing button-alloy data on Ta-8W-8Re (later evolved into T-111 and T-222).

(4) National Research Corporation was producing Ta-10W by double consumable-electrode arc melting. Their ingot size was 6 inches in diameter.

Tungsten

(1) Fansteel was producing unalloyed and doped tungsten rod and wire. Through isostatic pressing and furnace sintering, they could produce billets up to 200 pounds.

(2) Sylvania could produce tungsten ingots, 7-3/4 inches in diameter, primarily for wire and massive pieces. They had not produced any alloy sheet.

(3) Firth Sterling primarily had experience with massive tungsten, and hoped to work on tungsten sheet, primarily of the thoriated-tungsten type. Their maximum billet size by vacuum sintering was 10" in diameter.

(4) Westinghouse (Bloomfield Works), an old-line producer of tungsten wire for the lamp industry, also had experience with isostatic-pressed and furnace-sintered ingots, and, in fact, pioneered this process. Their alloy interest was largely based on thoriated tungsten, but they also were interested in additions of carbides, such as W-0.38TaC.

Overall Status

At the conclusion of the presentations, the members of the Alloy Requirements & Selection Subpanel each commented on their reactions. Some of the individual reactions are rather revealing in the context of subsequent developments in the industry. A few of these are indicated below:

The activity of the refractory metal field is at an amazing level considering the (small) potential size of the market.

Was surprised to see so many companies in the field.

Overall, there has been a great deal of alloy development and this is rather fantastic considering the market.

Was encouraged at the large amount of advanced furnace and fabrication facilities being installed by producers.

The stories on coatings were meager and apparently over-optimistic.

Chemical analysis needs attention.

There are little data on quality and reproducibility.

Alloys of promise presented were Mo-0.5Ti, Mo-0.5Ti-0.07Zr, Mo-SiO₂, F-48, D-41, Co-5Mo-5V, Co-5Mo-5V-1Zr, Ta-10W, unalloyed tungsten, and W-ThO₂.

Overall, it appeared to the Alloy Requirements & Selection Subpanel that the producers of refractory metals overestimated the market and underestimated the technical problems in refractory metals. The processing technology for molybdenum was reasonably well established, and molybdenum alloy development was advanced to the point where reasonably well-optimized alloys could be selected. Ingot and mill processing procedures for columbium and tantalum alloys were in a very undeveloped state. Many of the columbium alloys that looked promising in 1959 subsequently have been discontinued. Tantalum-alloy development was in its infancy. The processing of tungsten sheet was not yet worked out. Most of the discussion of tungsten alloys stemmed from the use of thoriated tungsten in electron tubes.

Establishment of Technical Requirements

Applications

As a result of discussions and visits to organizations engaged in the development of advanced aerospace systems requiring refractory metals, a picture was evolved of the technical requirements. It appears that the advanced aerospace applications for which refractory metals are considered are primarily based on the resistance of refractory metals to aerodynamic heating, melting, and liquid-metal corrosion. These will be discussed in the following sections.

Aerodynamic Heating. Aerodynamic heating is involved in re-entry from orbit or outer space through flight corridors in which moderate heat fluxes are sustained for relatively long periods, compared to ballistic re-entry, e.g., the glide corridor projected for winged vehicles of the DynaSoar type. In such vehicles, weight is saved if the thermal-protection system employs refractory metals to radiate part of the heat. The choice of a refractory-metal thermal-protection system relative to an ablative system is enhanced if multiple re-entries are envisioned.

A second application for refractory metals involving resistance to aerodynamic heating is in large hypersonic vehicles, such as the aerospace plane or in hypersonic vehicles where low-altitude cruise is contemplated. In such systems, heat is sustained for a sufficiently long time that ablation cannot be considered as an alternative thermal-protection system because of the prohibitive weight penalties that would be incurred.

The refractory metal thermal-protection system might be of the hot-structure type in which the stresses are borne by the refractory metal structure itself. An alternative method is to use a cooled structure of nonrefractory metal separated from a refractory metal heat shield by insulation. In this case, the stress requirements are somewhat less demanding than for the hot structure, but the temperatures involved are higher, and there is equal need for high integrity, so that it is difficult to say whether the hot structure or heat shield concept entails the more difficult requirements.

The stresses to be withstood by the refractory-metal thermal-protection system are, to a considerable extent, at the option of the designer. It is to his benefit to use refractory metals of the maximum elevated-temperature strength possible. Since the duration of the re-entry may be relatively short, the structure might be designed on short-time tensile properties at elevated temperature. In sustained hypersonic flight, creep would be expected to be more important.

The aerodynamic heating application requires an oxidation-protection system. The coating choice may be influenced to a considerable extent by the refractory-metal substrate. Properly, the selection of a refractory metal for an aerodynamic heating application should not be divorced from the coating, since both, together with the proposed environment, comprise a system. However, because of the undeveloped state of coating technology, it proved impractical to consider coating performance in the selection of refractory-metal requirements.

Most thermal-protection systems involving refractory metals involve relatively thin-gage sheet of high quality relative to flatness

and gage. Sheet size was of less importance, since, in many cases, it was desirable to restrict component size to minimize thermal stress. The desired method of assembly of the refractory-metal structure would be through fusion welding, since a lighter weight structure would result. Welds in such fusion-welded assemblies should have some ductility to withstand residual and assembly stresses.

Containment of Flames. The use of refractory metals in nozzles for solid propulsion rockets is primarily dictated by melting-point consideration. The stresses on the flame side of the nozzle tend to be relatively small, but severe thermal stresses develop on the cold back side.

The choice of refractory metal in nozzles for solid rockets has switched from molybdenum to tungsten as the flame temperatures rose from about 5000°F to above 6000°F. The addition of metallic elements, such as aluminum, to the propellants results in an erosive condition, which is apparently resisted by molybdenum and tungsten, but not tantalum, although the addition of tungsten to tantalum improves this characteristic. Tantalum in solid rocket inserts has turned out to be secondary to tungsten.

The type of nozzle insert also has gone through evolutionary phases. For a considerable time, there was strong interest in sheet-metal liners cemented to graphite backings. However, firing tests have shown that such inserts tend to be unreliable, and often buckle as a result of the thermal expansion of the metallic liner relative to the graphite backing. Thus, the incentive for development of large tungsten sheets for nozzle liners was eliminated in favor of forged or infiltrated tungsten liners with relatively heavy sections.

A long-range possibility for the use of sheet-refractory-metal liners is with liquid-cooled liners. These have been made of both tungsten and Ta-W sheet alloys, but the development has not been pursued because of the phenomenal success of forged and infiltrated tungsten liners.

In a number of places, such as blast-tube liners and nozzle inlets, sheet tungsten finds use in solid rockets. However, the mechanical-property requirements for such service are relatively modest compared with the requirements for good fabricating characteristics.

Liquid Metal Containment. The third major category for considering refractory metals is the space-power-system application where it appears that a considerable amount of refractory-metal tubing would be needed. These technical requirements are covered in detail in the report prepared by the Refractory Metal Tubing Subpanel. Relatively little refractory-metal sheet will be needed in this application.

Overall Summary. The chief aerospace applications requiring refractory metal sheet alloys are involved in thermal protection against aerodynamic heating in manned re-entry and hypersonic vehicles. It was on this application that the technical requirements for the Refractory Metals Sheet Rolling Program were based.

AIA Survey of Refractory Metal Requirements

At the request of the Refractory Metals Sheet Rolling Panel, the Aerospace Research & Testing and the Propulsion Working Committees of AIA were polled in mid-1963 with regard to the present and future firm applications for refractory metals as well as the degree to which the Refractory Metals Sheet Rolling Panel was responsive to current and future needs and interests. Results of the survey are tabulated on the following pages.

TABLE 1

A. Engine Manufacturers (16)		
(1)	Definite requirement beyond experimentation	- 12
	Requirement for experimentation	- 1
	No requirement	- 3
(2)	Applications	
	Rocket motor vector control	- 2
	Control valves	- 1
	Nozzles	- 2
	Ion engines	- 2
	Gas turbines	- 5
	Liquid metal piping	- 3
(3)	Continue research and development on refractory metal sheet?	
	Yes	- 13
	No	- 3
	Waive	- 0

(4) Tubing requirement	
Yes	- 12
No	- 4
Waive	- 0
(5) Problems	
Oxidation	- 12
Fabrication	- 8
Joining	- 10
Brittleness	- 8
Reliability	- 4
Strength	- 3
Liquid metal corrosion	- 2
Fatigue	- 1
Thin metal sandwich	- 2
Recrystallization temperature	- 2
B. Airframe Manufacturers (25)	
(1) Definite requirement beyond experimentation	
Yes	- 15
No requirement	- 6
Requirement for experimentation	- 1
Waive	- 3
(2) Applications	
Nozzles	- 2
Radiation cooled motor	- 2
Re-entry surfaces	- 5
Aerospace vehicle	- 7
Recoverable booster	- 3
(3) Encourage more research and development on sheet?	
Yes	- 18
No or waive	- 2
(4) Tubing requirement	
Yes	- 13
No or waive	- 7
(5) Problems	
Brittleness	- 8
Thin metal sandwich	- 3
Fabrication	- 9
Low modulus of columbium	- 1
Recrystallization temperature	- 4
Coating and oxidation protection	- 13
Strength weight	- 10
Joining	- 12

(5) Problems (continued)

Flatness	- 1
Reliability	- 1
Cost	- 2
Weight	- 4

C. Overall Conclusion

The aerospace industry has extensive needs for refractory metals and endorses the Refractory Metals Sheet Rolling Panel.

In reviewing the AIA poll, the Alloy Requirements & Selection Subpanel noted a lack of specificity in the "firm" requirements, except for a few cases like X20 re-entry (since cancelled), ASSET, and solid-propellant nozzles. Thus, they felt that many of the "yes" responses were really "yes-if" responses, dependent on securing system contracts. Thus, there undoubtedly was a considerable degree of redundancy in the response. However, the Alloy Requirements & Selection Subpanel was encouraged by the high level of interest shown and the endorsement of the Refractory Metals Sheet Rolling Program. Certainly, refractory metals have emerged as a clear requirement for advanced aerospace systems.

Ground Rules

To qualify an alloy for consideration as a candidate for the Refractory Metals Sheet Rolling Program, the Alloy Requirements & Selection Subpanel established minimum development status. Since it was recognized that refractory-metal alloys were in many cases not highly advanced, support for pilot development as well as preproduction development was recognized. The following minimum billet or sheet sizes were to have been produced in order to qualify for these classes:

(1) Pilot Development Status

Ingots or billets:	2-inch minimum cross section
Sheet:	6 x 20-inch minimum size

(2) Preproduction Development Status

Ingots or billets:	6-inch minimum cross section
Sheet:	18 x 48-inch minimum size

A number of exceptions to the general ground rules were recognized as being necessary at the outset. In order to consider the possibility

of electron beam melted columbium or tantalum alloys, which had a maximum 5" diameter capability at the time, 5" section ingots and 12" x 36" sheets were taken as corresponding to preproduction status. In tungsten, because of the primitive state of sheet development at the time, a sheet size of 6" x 20" was taken as corresponding to preproduction as well as pilot status. Sheet gages were taken as 0.010" and 0.100".

Although disclosure of composition was not mandatory during presentation of an alloy candidate, producers were informed that disclosure would have to be made if the alloy was recommended for the sheet rolling program.

Targets

After the current and future applications for refractory metals and capabilities of state-of-the-art alloys were reviewed, various targets were set for refractory metal sheet according to the following format:

- (1) Fabricable molybdenum alloy
- (2) High-strength molybdenum alloy
- (3) Fabricable columbium alloy
- (4) High-strength columbium alloy
- (5) Pure tungsten
- (6) Alloyed tungsten
- (7) Tantalum alloys

(Table 2 gives the targets for refractory metal alloy selection as presented to the main panel on March 1, 1960.)

Basis for Targets

It was apparent from discussions with users of refractory metals that both low strength as well as high-strength materials would be useful. Where relatively low strength at elevated temperature was acceptable, very high quality and producibility were generally desired. Therefore, it was decided to set up targets for two types of molybdenum and columbium alloys. The first type would be an alloy which was readily fabricable to wide, thin sheet of high quality, which would be easily formed and joined by fusion welding. The second class of alloys would be those which were quite difficult to fabricate, but had considerably higher strength and recrystallization characteristics at elevated temperatures. Tungsten appeared to be the only refractory metal of interest in unalloyed form. The tungsten

TABLE 2. TARGETS FOR ALLOY SELECTION

Requirements	Fabricable Molybdenum		High-Strength Molybdenum		Fabricable Columbium		High-Strength Columbium	
	In Optimum Condition	Compl. Recryst.	In Optimum Condition	Compl. Recryst.	In Optimum Condition	Compl. Recryst.	In Optimum Condition	Compl. Recryst.
Room-Temperature Tensile								
Ultimate Tensile Strength, ksi	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)
Yield Strength, 0.2% Offset, ksi	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)
Elong., per cent	10	10	2	2	15	15	10	1
Elevated-Temperature Tensile								
Temperature, F	Optimum Condition	2400	Optimum Condition	3000	Optimum Condition	2400	Optimum Condition	2600
Ultimate Tensile Strength, ksi	75	50	75	25	50	20	50	20
Yield Strength, 0.2% Offset, ksi	60	35	60	15	40	15	40	15
Elong., per cent	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)
Creep Rupture (State Stress and Elong.) at								
Temperature, F	2000	2400	2400	3000	2000	2400	2200	2600
Rupture Time, hr	1 10	1 10	1 10	1 10	1 10	1 10	1 10	1 10
Recrystallization (In optimum condition)								
50% by met. obs.								
Time	1 Hr		1 Hr		1 Hr		1 Hr	
Temperature, F	2600		3200		2400		2800	
Notch-Sensitivity - Ratio^(b)								
	1.0 (RT)		1.0 (200 F)		1.1 (RT)		1.0 (RT)	
Transition Temperature (In optimum condition)								
In bending (4T)	-40		RT		-100		-40	
Tensile, notched	State		State		State		State	
smooth	"		"		"		"	
Impact, Charpy	"		"		"		"	
Room-Temperature, Bend Ductility								
Base metal	1T		4T		1T		4T	
Welded (Weld transverse to bend axis)	4T		State		2T		6T	
STATE FOLLOWING:								
Density								
Melting Point								
Emissivity								
Modulus of Elasticity								
Thermal-Shock Resistance								
Creep Properties								
Oxidation Resistance and Contamination								
Coatability								
Experience with 45° Brittleness								
Lamination Tendency								

(a) To be furnished

(b) K_t 6.0, RA 40% (See ASTM Bulletin, January 1960, p 29).

TABLE 2. TARGETS FOR ALLOY SELECTION

Pl. st.	Fabricable Columbium		High-Strength Columbium		Unalloyed or Dilute Tungsten		High-Strength Tungsten			Tantalum		
	In Optimum Condition	Compl. Recryst.	In Optimum Condition	Compl. Recryst.	In Optimum Condition	Compl. Recryst.	In Optimum Condition	Compl. Recryst.		In Optimum Condition	Compl. Recryst.	
	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)		(a)	(a)	
	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)		(a)	(a)	
2	15	15	10	10	2	(a)	2	(a)		15	15	
n	Optimum Condition		Optimum Condition		Optimum Condition		Optimum Condition			Optimum Condition		
0	2000	2400	2200	2600	3000	3500	3000	3500	4000	2400	3000	3500
5	50	20	50	25	20	10	36	27	15	35	25	15
5	40	15	40	15	15	7	24	18	10	28	16	10
	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)	(a)
0	2000	2400	2200	2600	3000	3500	3000	3500		2400	3000	
0	1 10	1 10	1 10	1 10	1 10	1 10	1 10	1 10		1 10	1 10	
	1 Hr		1 Hr		1 Hr		1 Hr			State		
	2400		2800		State		3400					
	1.1 (RT)		1.0 (RT)		1.0 (400 F)		1.0 (400 F)			1.2 (RT)		
	-100		-40		+300		+300			-320		
	State		State		State		State					
	"		"		"		"					
	"		"		"		"					
	1T		4T		4T (300 F)		4T (300 F)			1T		
	2T		6T		State		State			2T		

alloy, which appeared to be most advanced, was the nonmetallic dispersion-hardened type represented by thoriated tungsten. The high-strength tungsten target was based on data from such material. Since the time of setting the tungsten alloy target, it has become apparent that much more ductile tungsten alloys could be available than are suggested by the tungsten targets. These are the rhenium-containing tungsten alloys. The relatively meager amount of information available on tantalum-base alloys indicated that these alloys would have excellent fabricability as well as high strength at elevated temperature, and there was no need to set separate targets for fabricable and high-strength tantalum products. Also, it was felt that if a tantalum alloy were to be used, it should not be at the sacrifice of fabricability, including good weld ductility.

Comparison with AAAP Requirements

The short-time strength requirements for refractory metals suggested by the AAAP* appear to be fairly well met by RMSRP targets for high-strength refractory metals. Although there are few data available on creep and rupture properties of refractory metals (there are no RMSRP creep and rupture targets), it appears that the RMSRP alloys barely meet AAAP creep and rupture requirements in lower temperature ranges, and probably will fall far short in the higher temperature ranges. Also, it is apparent that the RMSRP alloys are responsive to the various individual but not to the combination of target properties of the AAAP report.

A comparison was made between the tensile target properties (not compensated for density) given in the AAAP report and the RMSRP targets for high-strength alloys. The RMSRP targets agree remarkably well with the AAAP targets, despite the fact that there was no exchange between the groups in setting up the targets. The AAAP targets were based on properties designers thought they would need, while the RMSRP targets were based on what materials people thought could be achieved.

*MAB Aircraft & Astronautics Applications Panel, MAB-139-M(AA3). Phase II Report (Classified), July 31, 1961.

Recommendations

In order to understand the seemingly aimless and contradictory course of the Alloy Requirements & Selection Subpanel in their considerations of refractory metals, it is necessary to realize that the alloys and processes were being developed and modified as the program proceeded through the years. Thus, the information will be presented through the summary tables in compliance with ARS3 targets and recommendations made at the various meetings where presentations were made.

Recommendation of Uniform Evaluation

May 20, 1960

It is recommended that, in the future, provision be made for uniform evaluation of refractory metal alloy sheet candidates nominated by various producers to determine their compliance with target properties.
(Recommendation implemented).

August 24, 1960

The conditions for uniform evaluation and tests agreed upon were set as given below:

- (1) The material evaluated should have been produced in a sheet of the size meeting the ground rules, and be of a thickness between 0.040" and 0.062".
- (2) Triplicate tensile tests, longitudinal and transverse, should be conducted at room temperature, at a strain rate of 0.003 to 0.007 per minute to yield, and 0.08 to 0.12 per minute to fracture.
- (3) Elevated-temperature tensile tests in the longitudinal direction should be conducted at 2000°F and 2400°F in a vacuum of 0.1 micron Hg or better.
- (4) Rupture tests should be conducted at 2000°F and 2400°F within a range of 1/3 to 1/2 of the ultimate tensile strength to establish the 1-hr and 10-hr rupture stress. The rupture specimen should be wrapped in tantalum foil, and the vacuum should be 0.1 micron Hg or better. The specimen should be soaked at least 1/4 hr before testing. The Knoop hardness

gradient across a section of the specimen should be determined after rupture testing to ascertain absence of contamination.

(5) The recrystallization temperature of the as-received material should be determined after heating in vacuum for 1 hr at successively higher temperatures. Microscopic examination and hardness changes should be used to estimate the temperature for 50 per cent recrystallization.

(6) The longitudinal and transverse bend ductility should be determined on specimens, about 1" x 3", at a head speed of about 10" per minute, using a male die punch with various nose radii in accordance with the procedures recommended by the Titanium Sheet Rolling Program (MAB-137-M). Sufficient specimens should be tested so that the minimum bend radius is determined to within 1T, using duplicate specimens.

(7) The bend transition temperature should be determined within a bracket of 50°F, using a male die with a 4T bend radius. Longitudinal specimens should be used.

(8) The weld ductility should be determined on 1" x 3" specimens with the weld normal to the bend axis and the weld bead ground flush. The specimen should have an actual weld joint, not bead on plate. If filler material is used, it should have the same composition as the base metal.

(9) Thermal stability should be determined from the residual bend ductility at room temperature after a 10 hr, 2400°F unstressed exposure in vacuum. Duplicate longitudinal bend specimens should be used.

(10) Oxidation tests will be conducted by exposing test coupons for 10 hrs at 2400°F in moving air. Metal loss and extent of internal contamination will be measured. Contamination will be indicated by a Knoop hardness transverse across a section of the exposed specimen.

(11) Lamination tendency will be determined by reverse bending duplicate specimens bent through the minimum bend radius and observing laminations, if any.

(12) A chemical analysis will be conducted for nominal alloying elements and interstitials (C, N, O, and H).

Molybdenum Alloys

May 20, 1960 (See Table 3)

(1) It is recommended that only one arc cast molybdenum sheet alloy in addition to Mo-0.5Ti be included in the Refractory Metals Sheet Rolling Program. This second alloy is Mo-0.5Ti-0.08Zr. As the second alloy for the Universal-Cyclops program, a comparison of two arc-cast molybdenum sheet alloys by a single producer will thus be provided.

(2) It is recommended that a second producer for the Mo-0.5Ti-0.08Zr alloy be provided to give a comparison from producer to producer for a single alloy. On the basis of the information presented to the group, a second producer should be Fansteel Metallurgical Corporation or Climax Molybdenum Company.

(3) No arc-cast molybdenum candidates of the high strength class were found sufficiently advanced in sheet form to warrant support by a sheet rolling program. Mo-1.5Cb, Mo-25W-0.12Zr, and Mo-1.25Ti-0.15Zr-0.15C are examples of high strength molybdenum alloys of considerable interest. It is recommended that those organizations that presented these alloys as candidates qualify them in sheet form and obtain data on them in conformance with the target property of the Alloy Requirements & Selection Subpanel.

November 30, 1962

(1) The Alloy Requirements & Selection Subpanel was impressed by the great progress that had been made in the quality of high strength molybdenum sheet alloys produced by the arc cast and powder metallurgy methods.

(2) A considerable amount of development work is in progress on both arc-cast and powder metallurgy alloys, indicating that even more interesting alloys may be forthcoming in the future.

(3) The Navy apparently plans to support the development of high strength molybdenum alloys of the TZC type at the 4" diameter ingot level.

TABLE 3. COMPLIANCE OF MOLYBDENUM-ALLOY SHEET CANDIDATES WITH
FABRICABLE MOLYBDENUM-ALLOY TARGET PROPERTIES

(As Recommended at the May 20, 1960 Meeting.)

Property	Target	Fansteel/Marquardt (TZM-0.5Ti-0.07Zr)	Climax (0.5Ti-0.08Zr)	Climax (0.05Zr)	General Electric (0.5Zr)
Room-Temperature Tensile Optimum	f_{tu} - f_{ty} -10(a)	152-134-12	135-121-10	117-104-13	110-88-15
Recrystallized	f_{tu} , f_{ty} -10	97-72-17	64-62-1	71-62-42	90- -30
Elevated-Temperature Tensile					
2000 F	75-60-(b)	74-65-5	43	20	54-48-9
2400 F	50-35-(b)	48-40-8			41-36-12
Stress-Rupture Strength, ksi					
2000 F	--	No data	No data	No data	--
1 Hr	--				--
10 Hr					
2400 F	--				32
1 Hr	--				23
10 Hr					
50% Recrystallized Temperature, F (1 Hr)	2600	2500	2500	2350	2550
Room Temperature Notch - Sensitivity Ratio	1.0	No data	No data	No data	1.1
Transition Temperature, F (4T)	-40	<R. T.	72-300 F	-50/50 F	<R. T.
Room-Temperature Bend Ductility					
Base	1T	1T	2T	1T	0.4T
Weld	4T	?	?	?	?

(a) Ultimate strength in ksi, 0.2 per cent offset yield strength in ksi, and per cent elongation in 1 inch.

(b) To be furnished.

It is also understood that the Manufacturing Technology Laboratory of the Air Force plans to support additional work aimed at solving the high strength molybdenum ingot cracking problem, at sizes of about 4" in diameter.

(4) The TZM alloy (Mo-25W-0.1Zr-0.03C) of Climax did not appear to the Alloy Requirements & Selection Subpanel to show sufficient advantage over the TZC (Mo-1.25Ti-0.3Zr-0.1C) type of high-strength molybdenum alloy to warrant sheet rolling program support.

(5) The Army Materials Research Agency at Watertown was authorized to evaluate TZC (Mo-1.25Ti-0.3Zr-0.15C) and MTC (powder metallurgy Mo-0.5Ti) sheet submitted.

(6) Since it appeared that those things which needed doing to permit further judgment of high-strength Mo sheet were, indeed, being done, further action by the Alloy Requirements & Selection Subpanel was deferred until Spring 1963, at which time the status and progress of both arc cast and powder metallurgy alloys will be reviewed.

April 17, 1963 (see Table 4)

(1) Climax TZC alloy, produced by arc melting, extrusion, and rolling, met the ground rules for pilot production and could be considered as a candidate for preproduction sheet rolling status. It is apparent that Climax has made excellent progress in producing a Mo alloy with high strength, heat treatability, and low transition temperature, with good prospects for production-type producibility. The elevated temperature strength of arc melted TZC with conventional processing corresponds to the fabricable rather than the high-strength class.

(2) A sheet of Climax TZC alloy at its present stage of development is recommended to be evaluated by AMRA. If this evaluation bears out the present favorable indications of the Climax data, the arc cast TZC Mo alloy would be recommended for a preproduction-type contract.

(3) It is understood that Climax is working under a BuWeps contract to develop optimum processing for TZC. When this work has proceeded to the point that optimum processing sheet is available, sheet material so processed should also be evaluated by AMRA for uniform evaluation.

TABLE 4. HIGH-STRENGTH MOLYBDENUM-ALLOY CANDIDATES

(Presented at April 17, 1963, Meeting)

Property	Targets		Climax TZC (1.25Ti-0.3Zr-0.15C) (85.4-99.6)-?-(4-29)	Sylvania TZM (0.25Ti-0.1Zr-0.01C) --	Climax TZM (0.5Ti-0.1Zr) (Not a candidate; for comparison)
	Fabricable Molybdenum	High-Strength Molybdenum			
Room-Temperature Tensile(a)					
Optimum	(b) ₁₀	(b) ₂	(125.0-150.4)-?-<8	131-117-14	10-(b) ₁₀
Recrystallized	(b) ₁₀	(b) ₂	(65.4-99.6)-?-(4-29)	--	--
Elevated-Temperature Tensile(a)					
2000 F	75-60-(b)	--	--	--	--
2400 F	50-35-(b)	75-60-(b)	(49.2-51.8)-?->	(54-61)-(48-53)-(7-12)	42-(b) ₁₀
3000 F	--	25-15-(b)	(10.2-10.4)-?->	(9-10)-(5-8)-(61-59)	10-(b) ₁₀
Stress Rupture Strength, ksi					
Temperature, F					
10 Hr	2000	2400	2400 3000	2400	2400
	(b)	(b)	21.0 6.9	18	22
50% Recrystallization Temperature, F (1 Hr)	2600	3200	3000 (100%)	--	2500-2700 (100%)
Transition Temperature, F (4T)	-40	R.T.	-10(L), 50(T)	-13 to 32	-100(L), -30(T)
Room-Temperature Bend Ductility					
Base	1T	4T	2T	4T	2T
Weld	4T	State	?	?	?

(a) Ultimate strength in ksi, 0.2% offset yield strength in ksi, and per cent elongation in 1 inch.

(b) To be furnished.

(4) The Manufacturing Technology Laboratory of the Air Force is considering a program on cracking in large Mo ingots. It is recommended that the MTL contract, when awarded, should cover the TZC alloy, or its equivalent.

(5) Sylvania has encountered considerable difficulty in producing powder metallurgy TZC Mo alloy, and was not in a position to recommend the alloy.

(6) The powder metallurgy TZM molybdenum alloy produced by Sylvania appears to have an elevated temperature strength advantage over arc-cast TZM. In fact, the powder metallurgy TZM has elevated temperature strength comparable to that of arc-cast TZC. The producibility demonstration of powder metallurgy TZM was limited, however. Since high-strength class Mo alloys were being considered, it was decided not to make any recommendations on the Sylvania TZM candidate.

Columbium Alloys

July 7, 1960 (see Table 5)

(1) It was concluded that most of the alloys presented to the Alloy Requirements & Selection Subpanel on July 6 and 7, 1960, were not sufficiently developed to warrant selection at the present time.

(2) It was recommended that du Pont D-41, one of the Temescal alloys, Union Carbide CB-74, General Electric A8-55, and Fansteel Cb-Ta-W-Zr alloys be provided by the producers to Watertown Arsenal Laboratory in sufficient quantities to permit uniform evaluation.

(3) Columbium alloys no longer being considered for alloy selection are Fansteel 82-B, General Electric F-48, and du Pont D-31.

(4) It is recommended that selection of a columbium-sheet alloy be deferred until November 1960.

(5) Evaluation of the various alloys presented on July 6 and 7, 1960, were as follows:

TABLE 5. COMPLIANCE OF COLUMBIUM SHEET ALLOY CANDIDATES WITH TARGET PROPERTIES

(July 7, 1960, Meeting)

Property	Targets				Temescal (Cb-Ta-W-Mo-Zr)	Union Carbide (CB-74)	General Electric (F-48)	Fansteel	
	Fabricated Columbium	High-Strength Columbium		Du Pont				828	Cb-Ta-W-Zr
Room Temperature Tensile Optimum Recrystallized	f_{tu} , f_{ty} , 15(a)	f_{tu} , f_{ty} , 10	100-95-15	130-127-8	ND	89-79-27 (Bar)	122-111-16	90-80-15	110-99-15
	f_{tu} , f_{ty} , 15	f_{tu} , f_{ty} , 10	96-85-35	127-118-12	ND	88-70-30 (Bar)	ND	65-50-20	74-54-19
Elevated Temperature Tensile	50-40-(b)	--	30-24-25	--	54-47-15	51-47-18 (Bar)	--	--	68-64-14
	--	50-40-(b)	--	49-47-20	--	32-30-31 (Bar)	40-37-20	33-23-18	35-30-30
	20-15-(b)	--	10-10-15	34-29-3	35-31-10	--	--	--	18-17-56
	--	25-15-(b)	--	--	32-29-10	--	--	--	--
Stress-Rupture Strength, ksi Temperature, F	2000, 2400	2200, 2600	2000	2000	ND	ND	2200	ND	2200
	(b) (b)	(b) (b)	-- 11	-- 23	ND ND	ND ND	-- 25	ND ND	24 (6 hr)
50% Recrystallization Temperature, F (1 Hr)	2400	2800	21-2300	2400	ND	2400	2500	2100	ND
Notch-Sensitivity Ratio	1.1 (R.T.)	1.0 (R.T.)	ND	ND	ND	ND	0.85-1.0	ND	ND
Room-Temperature Transition Temperature, F.	-100	-40	-320 to 40	ND	ND	ND	<-100	ND	ND
Bend Ductility Base Weld	1T 2T	4T 6T	2T Limited	3T ND	1T ND	ND ND	0.4 Brittle	1T >1T	1T 1T
	(b)	(b)	8.08	8.6	ND	8.6	9.4	10.26	10.48

(a) Ultimate strength in ksi, 0.2 per cent offset yield strength in ksi, and per cent elongation in 1 inch.

(b) To be furnished.

(a) du Pont D-31 should be eliminated from further consideration as not having as good potential as du Pont D-41. Also, it appears that the D-31 alloy will be selected as the second columbium alloy in the Air Force columbium sheet-rolling program.

(b) du Pont D-41 is of continuing interest. However, it is not yet far enough along in sheet form to permit estimation of its probable producibility, or comparison with target properties.

(c) Temescal Cb-W-Ta-Mo-Zr alloys. Very little evaluation data were provided on this group of alloys. They are of continuing interest because of their cold-rollability and attractive strength at elevated temperatures. A composition still has to be fixed and more evaluation data obtained.

(d) Union Carbide CB-74 is of continuing interest to the Subpanel. When sufficient sheet has been rolled to qualify for ARSS ground rules, and evaluation data in compliance with the target properties are available, it should be further considered.

(e) General Electric F-48 was eliminated from further consideration because of the fabrication difficulties which have been associated with this alloy. Its elevated-temperature strength did not appear to show advantage over more fabricable alloys. Weld and recrystallization embrittlement are serious disadvantages. The alloy is in the Air Force sheet-rolling program, and will be evaluated in any case.

(f) General Electric AS-55 is of potential interest to the Subpanel as a fabricable high-strength columbium alloy. After compliance with ground rules and targets are available, it should be further considered.

(g) Fansteel FS-82-B was eliminated from further consideration as not being so attractive as Fansteel FS-83, Cb-Ta-W-Zr alloy.

(h) Fansteel Cb-Ta-W-Zr alloy is of continuing interest to the Subpanel as a fabricable, weldable, high-strength columbium sheet alloy. Its elevated-temperature strength is particularly noteworthy in view of its excellent fabricability. It appears among the most attractive of the alloys presented thus far.

March 1, 1961 (see Table 6)

(1) It is recommended that proposals be solicited from the Fansteel Metallurgical Company on their Cb-24Ta-10W-1Zr sheet alloy and from Union Carbide Metals Company and Haynes-Stellite on their Cb-10W-5Zr sheet alloy for development under the Navy Department Refractory Metals Sheet Rolling Program from their present pilot status to preproduction status.

(2) The proposals should contain technical information pertinent to the alloy candidates in compliance with the target properties, including any supplementary information to that originally presented. In particular, information on fusion weldability and fabrication characteristics is desired. These proposals will be considered by the Navy Department with the Alloy Requirements & Selection Subpanel, and a final selection made between the two alloy candidates for recommendation.

(3) Further consideration of columbium alloys will be held through a review of the columbium alloy situation in about 6 months.

(4) The evaluations provided were disappointingly incomplete, and it is hoped that, through the uniform evaluation at Watertown Arsenal Laboratory, this situation can be alleviated.

August 8, 1961 (see Table 7)

(1) The Alloy Requirements & Selection Subpanel concluded that the two alloys (Fansteel FS-85, Cb-27Ta-12W-1Zr, and Union Carbide Cb-752, Cb-10W-4Zr) were at a standoff with respect to the columbium sheet target properties, but that the Fansteel alloy had greater potential for producing high-quality sheet. The Fansteel alloy, therefore, was recommended for support.

(2) It was further recommended that, if possible, there should be a second producer for the Cb-Ta-W-Zr alloy, and that consideration should be given to organizations with advanced fabrication facilities, particularly designed for refractory metals, like Haynes-Stellite or du Pont, as the second producer.

TABLE 6. COMPLIANCE OF COLUMBIUM

(Presented at March)

	Target		General Electric		Du Pont				
	Fabrication	High Strength	AS55	F48	D31S	D31	D41	D99	
Type			Fab.	High strength	Fab.	Fab.	High strength	Fab.	Fab.
Composition	--	--	Prop.	15W-5Mo-1Zr-0.05C	Ti-10Mo-0.1C	10Ti-10Mo	10Ti-20W-8Mo	10Ti-5Zr	1Ti-15Hf
Max Ingot Diam, in.	--	--	4-1/2	6	3	8	3-3/4	3	2-1/2
Max Sheet Size, in.	--	--	1-1/2 x 15	14 x 60	9 x 12	25 x 31	5 x 10	10 x 20 6 coil	6 x 20
Room-Temperature									
Tensile									
Stress Relieved	$f_{tu}, f_{ty}, 15^{(a)}$	$f_{tu}, f_{ty}, 10$	76-66-14	122-111-16	142-124-14(B)	100-95-15	130-127-8	110-108-7	59-38-5
Recrystallized	$f_t, f_{ty}, 15$	$f_{tu}, f_{ty}, 10$	68-55-28	117-102-18	ND	96-85-35	127-118-12	80-70-21	ND
Elevated-Temperature									
Tensile									
2000 F	50-40-	--	34-32-26	--	40-37-47(B)	30-24-25	--	23-23-48	36-28-3
2200 F	--	50-40-	21-16-50	40-37-20	--	--	49-47-20	--	21-20-5
2400 F	20-15-	--	--	--	--	10-10-15	34-29-3	--	--
2600 F	--	25-15-	--	--	--	--	--	--	--
Stress-Rupture Strength									
Temp, F/time, hr	2000-2400	2200-2600	2000/10	2200/10	2000/10	2000/10	2000/10	ND	ND
Stress, ksi	--	--	30	25	>10 <14	11	23		
50% Recrystallization Temperature, F	2400	2800	2300	2500	<2700(100%)	21-2300	2400	ND	<2550
Notch Tensile ^(c)	1.1(R. T.)	1.0(R. T.)	ND	0.85-1.0	ND	ND	ND	ND	ND
2T Bend Trans., F	-100	-40	ND	<-100	<200(L)>80(T)	-320 to 40	ND	<-320	ND
Bend Ductility									
Base	1T	4T	<3T	0.4T	2T	2T	3T	"V good"	ND
Weld	2T	6T	<4T	Brittle	ND	"Limited"	ND	"V good"	2T(200)
Density, g/cc	--	--	--	9.4	8.08	8.08	8.6	7.0	8.5

(a) Ultimate tensile strength in ksi, 0.2 per cent offset yield in ksi, and per cent elongation in 1 inch. (B) refers to bar data, otherwise data are

(b) Data provided by General Electric.

(c) ASTM edge-notched sheet specimen, 30-40 per cent notch depth, less than 0.001-inch notch radius. (ASTM Bulletin, January, 1960, p 29.)

Previous pages were blank, therefore not filmed.

OF COLUMBIUM-ALLOY CANDIDATES WITH TARGETS

(Presented at March 1, 1961, Meeting)

	Westinghouse				Wah Chang		Union Carbide	Stauffer Metals			
	B22	B55	B66	B77	C-103	C-120	(0374)	SCB-41	-61	-278	-291
	Fab.	Fab.	Fab.	Fab.	Fab.	High strength	Fab.	Fab.	Fab.	Fab.	Fab.
5Zr	1Ti-1Zr-5Hf	5V-5Mo	5V-5Mo-1Zr	10W-5V-1Zr	10Hf-1Ti-0.5Zr	(EB F48)	10W-5Zr	Prop.	Prop.	Prop.	Prop.
	2-1/2	3	3	3	3	4	4	1-1/2	1-1/2	3	5
20 oil	6 x 24	None	3-1/2 x 12	4 x 18	6 x 36	Unknown	15 x 15	6 x 24	6 x 24	6 x 36	6 x 36
8-7-21	59-38-22 ND	110-94-43 ND	113-88-31(B) ND	128-102-15(B) ND	93.5-88-9 56-42-33	-- (102-90-21) ^(b)	97-84-20 87-72-25	139-7 ND	136-7 ND	118-113-3 67-52-23	120-8 66-51-20
48	36-28-30 21-20-9 -- --	41-36-56(B) 28-26-60 -- --	54-46-38(B) 41-38-35 -- --	64-60-26(B) 43-39-49 -- 18-16-80	26-18-62 -- -- --	50-42-14 -- 26-19-38 --	52-45-8 36-31-16 -- --	66-57- 57-49- 45-39- 35-29-	59-51- 51-45- 41-35- 33-27-	-- -- -- --	-- -- -- --
	ND	ND	ND	ND	ND	ND	2000/10 33	ND	ND	2400/10 9	3000/10 4
	<2550	ND	ND	ND	1900	(2000) ^(b)	2400	2400	2400	ND	ND
	ND	ND	ND	ND	ND		ND	ND	ND	ND	ND
20	ND	ND	ND	ND	<-320		ND	ND	ND	ND	ND
rod"	ND	ND	ND	ND	0.5T	ND	<1T	Poor	Poor	"V good"	1T
rod"	2T(200 F)	ND	ND	2T(400 F)	0.5T	ND	ND				1(Zr) 100% efficient
	8.5	8.5	8.5	8.5	8.85	9.4	9.1	--	--	--	--

Other data are for sheet material. ND indicates no data supplied.

TABLE 7. COMPLIANCE OF FANSTEEL AND HAYNES STELLITE COLUMBIUM SHEET ALLOYS WITH RMSRP TARGETS FOR FABRICABLE COLUMBIUM ALLOYS

(As Considered at August 9, 1961 Meeting.)

Property	Target	Fansteel FS-86(a)		Haynes Stellite Cb-782(b)	
		(W 10.5-11.5, Ta 27.6-28.2, Zr 0.9-0.95, low interstitial)		(W 10, Zr 3.2-4.2, low interstitial)	
Room-Temperature Tensile Stress Relieved	f_{tu} , f_{ty} , 15(c)	105-95-14		102-87-12	
Recrystallized	f_{tu} , f_{ty} , 15	79-66-33		86-71-27	
Elevated-Temperature Tensile					
2000 F	50-40	46-41-13		47-37-8	
2200 F	--	33-31-27		32-27-22	
2400 F	20-15	23-20-40		30-27-35 (bar)	
Strength-Density ratio, 2000 F	--	118-105		145-114	
2200 F	--	85-80		99-83	
Stress-Rupture Strength, ksi					
2200 F - 10 Hr	--	19		20	
50% Recrystallization Temperature, F (1 Hr)	2400	2500		2400	
Notch-Sensitivity Ratio	1.1	ND		ND	
Transition Temperature, F	--	ND			
Tensile	--				Less than -107 F (stress relieved)
Bend	-100	Less than -320 F (recrystallized)		Less than -40 F (recrystallized)	
Room-Temperature Bend Ductility				Less than RT	
Base	--	Less than 1T		Less than 3T	
Weld	--	Less than 1T		Less than 3T	

TABLE 7. (Continued)

Property	Target	Fansteel FS-85(a)		Haynes Stellite Cb-752(a) (W 10, Zr 3.2-4.2, low interstitial)
		(W 10.5-11.5, Ta 27.6-28.2, Zr 0.9-0.95, low interstitial)		
Modulus of Elasticity, 10^6 psi	--	20		16
Density, lb/in. ³	--	0.39		0.32
E/d, 10^6 psi	--	51		49

- (a) 5-inch-diameter, 75-lb ingots, electron beam and arc remelted. Most sheet produced so far is 6 inches wide; some 13 inches wide also produced.
- (b) 6-inch-diameter, 350-lb ingots contemplated, electron-beam melted; most available data based on 4-inch-diameter e-b ingot. Previous sheet mostly 6 inches wide, one 15 x 10 inches; plan to produce 24 inches wide from larger ingots.
- (c) Ultimate tensile strength and 0.2 per cent yield strength in ksi and elongation in per cent. Fansteel used a 2-inch gage length, while Haynes Stellite used a 1 inch.

April 17-18, 1962 (see Table 8)

(1) Two solid-solution sheet alloy candidates, FS-85 (Cb-27Ta-10W-1Zr) and B-66 (Cb-5Mo-5V-1Zr) should be given a preliminary evaluation at the preproduction level, by having two preproduction ingots of each alloy prepared and fabricated by both Fansteel and Westinghouse.

(2) Evaluation of the preliminary material should be conducted by the producers themselves and by Watertown Arsenal Laboratory to provide the basis for a contract to one of the two producers on one of the two alloys.

(3) du Pont's X-110 (D-43) alloy apparently requires special processing to develop dispersion-hardening characteristics. It is understood by the Alloy Requirements & Selection Subpanel that du Pont will take over the Air Force columbium sheet rolling contract. If so, the ARSS strongly endorses X-110 as one of the alloys to be included in the contract. This endorsement will be tendered to the Air Force by the Materials Advisory Board.

(4) Watertown Arsenal Laboratory evaluation of Westinghouse B-66 and du Pont X-110 is not yet available. Implementation of the above recommendations will be contingent on the WAL check of the producer information.

(5) Independent evaluation of the weld ductility to confirm the producer information should be provided by an independent laboratory. Battelle Memorial Institute, through DMIC, was mentioned as a possible laboratory. The welding should be done under contamination-free conditions without use of filler other than the base alloy, on strip specimens at least 1" x 2", 0.062-inch minimum gage. Testing should be accordance with the Materials Advisory Board recommendation of 10 inches per minute with the bend axis perpendicular to weld.

The Alloy Requirements & Selection Subpanel evaluations of the alloys are summarized below:

- (a) Fansteel FS-85: The advantages of this alloy are good hot break-down and cold-finishing characteristics, weld ductility, and high-temperature strength. The alloy meets all the ARSS targets. However, its high density, resulting from its tantalum and

TABLE 8. COMPLIANCE OF COLUMBIUM-ALLOY CANDIDATES WITH FABRICABLE A

(Presented at April 17, 18, 1962, Meeting.)

Composition	Target	Fansteel FS-85 (Cb-27Ta-10W-1Zr)	Haynes Stellite CB-752 (Cb-10W-2.5Zr)	Du Pont (Cb-10W-1Zr)
Room-Temperature Tensile				
Stress Relieved	$f_{tu}, f_{ty}, 15^{(a)}$	109-92-14 (94-77-23) ^(b)	(82.6-89.7)-(64.3-73.6)-(18-28)	97-77
Recrystallized	$f_{tu}, f_{ty}, 15^{(a)}$	94-70-25	(95-79-20) ^(b)	77-2-
Elevated-Temperature Tensile				
2000 F	50-40-	55-43-20 (45-36-11) L, (50-42-10) T ^(b)	(43-50)-(29-39)-(22-38) ^(b)	47-40
2200 F	--	--	26-18-27	36-25
2400 F	20-15-	27-19-55 (21-10-35) L, (25-12-21) T ^(b)	(21-26)-(14-23)-(71-66) ^(b)	26-22
2600 F	--	13-15->80	--	--
Stress-Rupture Strength, ksi				
2000 F				
1 hr	State	30	--	31
10 hr	State	25 (est.)	(26) ^(b)	23
2200 F				
1 hr	State	--	--	--
10 hr	State	19	20	--
2400 F				
1 hr	State	16	--	--
10 hr	State	13	--	--
50% Recrystallization Temperature, F (1 Hr)				
	2400	2500 (Metallog.) 2100 (Mech. prop.)	2300	2400
Transition Temperature, F				
4T Bend	-100	<-320	<108, >-320 (4T) ^(b)	-100 (
Tensile	State	<-320	<32, >-40	--
Room-Temperature Bend				
Ductility				
Base	1T	1T	0T	1T
Weld	2T	4T	3T	<2T
Density, lb/in. ³	State	0.391	0.325	0.32

(a) Ultimate strength in ksi, 0.2% offset yield strength in ksi, and per cent elongation in 1 inch.

(b) Watertown Arsenal data (on CB-752 corresponds to 3.25Zr material).

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NIOBIUM-ALLOY CANDIDATES WITH FABRICABLE ALLOY TARGETS

(Presented at April 17, 18, 1962, Meeting.)

Haynes Stellite CB-752 (Cb-10W-2.5Zr)	Du Pont X110 (Cb-10W-1Zr-0.1C)	Westinghouse B-66 (Cb-5Mo-5V-1Zr)	General Electric AS-55 (Cb-5W-1Zr-0.06C-0.2Y) as Y ₂ O ₃
(82.6-89.7)-(64.3-73.6)-(18-28)	97-77-16	114-93-25	(75-109)-(65-95)-(14-23)
(95-79-20) ^(b)	77-?-12	107-82-34	68-55-28
(43-50)-(29-39)-(22-38) ^(b)	47-40-16	60-48-23	(30-40)-(25-38)-(25-15)
26-18-27	36-25-17	45-40-26	(24-30)-(16-28)-(24-50)
(21-26)-(14-23)-(71-66) ^(b)	26-22-28	30-29-30	--
--	--	--	--
--	31	--	--
(26) ^(b)	23	--	24
--	--	25	22
20	--	17	17
--	--	11	--
--	--	7	--
2300	2400	2300	2300
<108, >-320 (4T) ^(b)	-100 (2T)	<-320	-100
<32, >-40	--	<-320	--
0T	1T	1T	0.37T
3T	<2T	2.5T	4T(-100)
0.325	0.326	0.305	0.319

gation in 1 inch.

2

tungsten content, is disadvantageous, and reduces its high-temperature strength on the strength-weight basis.

- (b) Haynes-Stellite CB-752: This alloy has excellent cold-finishing characteristics. However, hot fabricating characteristics are reportedly poor, and extrusion and rolling in protective metallic packs appears necessary. Another concern is lowering the zirconium content from 5 per cent to 2.5 per cent, which is accompanied by a loss of high-temperature strength. The data available on low-zirconium CB-752 are meager, but evaluation of this material under the Air Force ASSET program is expected to provide this information.
- (c) duPont X-110: This is a promising dispersion-hardening alloy which meets all of the ARSS targets. Special processing is required to develop the desired fine dispersion of zirconium carbide. So far, the process has not been demonstrated beyond the 4-inch-diameter-ingot stage. It is understood that the dispersion-strengthening effect is lost as a result of welding or recrystallization.
- (d) Westinghouse B-66: This alloy has excellent cold-finishing characteristics and highest strength at elevated temperature of any of the candidates. Originally, it was reported to have poor weld ductility, but this has been reported to have been improved by reducing the interstitial content. There still remains some concern over its weld ductility, which will require independent confirmation of weld ductility.
- (e) General Electric AS-55: This is a dispersion-hardened alloy developed originally for tubing. Only laboratory-size sheet has been fabricated, and the alloy has not passed the ARSS ground rules for sheet. It is understood that NASA is supporting a study of processing of this alloy, and that further information on the properties of the sheet form, on at least a pilot level, will be forthcoming.

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- (f) Wah Chang C-129: This alloy has excellent fabrication characteristics but is not ductile in the as-welded condition, and was not further considered for alloy selection, which was primarily for the weldable alloys.

December 13, 1963 (see Table 9)

(1) The Alloy Requirements & Selection Subpanel decided to recommend FS-85 over the others categorized above on the basis of the alloy's excellent primary and secondary fabrication characteristics, including welding, and its excellent creep resistance. Since the processing schedule of FS-85 appears to be well worked out, with no problem areas, the Phase I evaluation should be limited to reproducibility characteristics and property evaluation.

(2) The B-66 alloy (Cb-5V-5MO-1Zr) was considered to have marginal fabricability. Its elevated-temperature property, particularly creep, offered no advantage over more fabricable alloys.

(3) C-129-Y (Cb-10W-10Hf-0.1Y) had equivalent producibility and ductility characteristics to FS-85, but its elevated-temperature strength potential was not as good.

(4) D-43 (Cb-10W-1Zr-0.1C) and CG-752 (Cb-10W-2Zr) are both presently in Air Force sheet rolling programs and were not considered in the recommendation for a Navy program other than for comparison purposes. Both alloys seem to be of the same type, with D-43 offering a better overall combination of processing, fabrication, ductility, and strength characteristics.

(5) Overall, the Alloy Requirements & Selection Subpanel recognized D-43 and FS-85 as two outstanding columbium sheet alloys to be supported by production sheet rolling programs.

(6) The Alloy Requirements & Selection Subpanel recommends to the Navy an evaluation-type sheet rolling program on Fansteel FS-85. Sufficient material should be produced, according to Fansteel's optimum schedule, to the MAB quality specification, for Phase I (Evaluation & Reproducible Demonstration), Phase II (Design Criteria), and Phase III (Component Fabrication).

TABLE 9. COMPLIANCE OF COLUMBIUM-ALLOY CANDIDATES WITH MAB FABRICABLE COLUMBIUM-ALLOY TARGETS

(A: Considered at December 13, 1963, Meeting.)

Property	Target	Fansteel FS-85 (Cb-27Ta-10W-1Zr) ^a	Westinghouse B-66 (Cb-8Mo-5W-1Zr) ^a	Wah Chang C125Y (Cb-10W-10Hf-0.1Y) ^a	Du Pont D43 (Cb-10W-1Zr-0.1C) ^a	Haynes Stellite Cb-752 : (Cb-10W-2.6Zr) ^a
Room-Temperature Tensile Stress Relieved	f_{tu} - f_{ty} -15 ^(a)	93.0-77.0-18(L) 83.8-83.0-15(T)	115.5-95.0-20	--	88-72-22(Opt)	85.3-69.8-37(Opt)
Recrystallized	f_{tu} - f_{ty} -15 ^(a)	84.4-64.3-23(L) 86.4-67.1-22.5(T)	102-77-33	91.6-76.7-20(L) 90.5-75.9-20(T)	--	81.9-63.1-27(L) 88.5-68.8-25(T)
Elevated-Temperature Tensile	50-40-10	45.6-43.5-22(SR)	52.0-42.5-20(Ext)	40.4-29.9-36	47-45-12	42-32-12(SR)
2000 F						
2400 F	20-15-EL	23.5-20.9-61(SR)	26.1-26.0-87(SR)	23-22-67(Ext) ^(b)	27-25-25	23-19-43(SR) 26.5-19.8-67(Opt)
Stress-Rupture Strength, ksi						
2000 F						
1 Hr	State	30	29	--	33	27
10 Hr	"	25	--	--	29	22
2200 F						
1 Hr	"	--	25	--	22	20
10 Hr	"	19	17	--	17	17
2400						
1 Hr	"	16	11	--	15	15
10 Hr	"	13	7	--	10	11
50% Recrystallization Temperature, F (1 Hr)	2400	2250	1900-2300 (Dep. on Red.)	2000	2400	2300
Transition Temperature, F						
4T Bend	-100	<-320	-100	<-320	-100(2T)	<-320
Tensile	State	<-320	<-320	<-320	<-80	<-32, >-40
Room-Temperature Bend						
Ductility						
Base	1T	<1T	<1T	<1T	1T	0
Weld	2T	<1T	3T	<1T	<3T	3
Density, lb/in. ³	State	0.391	0.305	0.343	0.325	0.325

(a) Ultimate strength in ksi, 0.2 per cent offset yield strength in ksi, and per cent elongation in 2 inches. (L) indicates longitudinal, (T) transverse.

(SR) stress relieved, (Rex) recrystallized, and (Opt) optimum condition in which the material was tested.

(b) (Ext) indicates data were interpolated from graphical data.

Tantalum Alloys

November 3, 1961 (see Table 10)

(1) Although certain of the candidate alloys showed promise by meeting most of those target properties for which data were provided, no alloy fully met the target properties. The Battelle Ta-30Cb-7.5V alloy substantially met target properties at 2400°F, and the Westinghouse Ta-8Hf-2W alloy at both 2400°F and 3000°F (based on undersized-ingot data). In no instance was all of the essential information provided. For these and other reasons, Refractory Metals Sheet Rolling Program support is not recommended at this time for any candidate alloy. (Comment: One of these alloys, Ta-30Cb-7.5V, will be supported by a U. S. Air Force sheet rolling project. Another, Ta-8W-2Hf, will be part of a U. S. Navy alloy scale-up and optimization program.)

(2) The alloy Ta-10W is considered by the Panel to have attained commercial status and does not require development support.

(3) Additional property data should be requested from Westinghouse, Stauffer, and Fansteel in order to enable consideration of their candidate alloys. It should be advised, however, that reported ductility values of the Stauffer and Fansteel candidates do not meet the target, and make their ultimate acceptance unlikely.

(4) The Ta-10Hf-5W alloy is considered too deficient in weldability to warrant support.

(5) Stauffer and Westinghouse are agreeable to supplying a representative sheet at least 2 square feet in area of their respective alloys: Ta-10W modified and Ta-8W-2Hf. These sheets will be forwarded to Watertown Arsenal Laboratories for testing marked for the attention of Mr. Thomas DeSisto. Battelle does not have material on hand, but will explore the possibility of providing for testing a like sample through Air Force and/or du Pont cooperation.

(6) The uncertain supply situation and the changing applications picture necessitate further information in order to determine the level for recommended support of tantalum-alloy development. Accordingly, data

TABLE 10. PROPERTIES OF CANDIDATE TANTALUM ALLOYS

(As Presented at November 3, 1961, Meeting)

	Target	Battelle (30Cb-7-1/2V)		Battelle (10Hi - 5W)		Wah Chang (10W)		National Res
		S. R.	Annealed	S. R.	Annealed			(10)
Room-Temperature Tensile, Stress-Relieved Condition								
Ultimate Tensile Strength, ksi		155	120	135	105	(200*)	98**	(180)
Yield Strength, 0.2% Offset, ksi		140	104	130	98	197	90**	(160)
Elong., per cent	15	15	28	15	25	4	12**	4
*Numbers in parentheses are c								
**Properties are stress-relieved								
Elevated-Temperature Tensile, Stress-Relieved Condition								
Temperature, F	2400 3000 3500 2400 3000 3500 2400 3000 3500							2200 F 2500 F
Ultimate Tensile Strength, ksi	35 25 15 33 9.5 -- 39 18 (11)							67 22
Yield Strength, 0.2% Offset, ksi	28 16 10 27 5.7 -- 31 15.7 (8)							55 20
Elong., per cent	-- -- -- 87 100 -- 55 51 (53)							4 22
Stress Rupture (State Stress and Elong.) at								
Temperature, F								
Rupture Time, hours	T							
1-Hr Recrystallization (In optimum condition)								
50% by met. obs.	State		2200 F		2400 F		2600	2500 F
Notch-Sensitivity Ratio	1.2T							
Transition Temperature (In optimum condition)								
In bending (4T)	-320		-320 F		<-320 F			
Tensile, notched			-320 F		<-320 F			
smooth								
Impact, Charpy								*Slightly above nitrogen temp
Room-Temperature Bend Ductility								
Base metal	1T		0T		2 - 4T		4T	
Welded (Weld transverse to bend axis)	2T		0T		Brittle			
STATE FOLLOWING								
Density, lb/in ³			9.429		0.599			
Melting point			4405 ± 90		5,114 ± 90			
Emissivity								
Modulus of Elasticity, 10 ⁶ psi			27		27			
Thermal-Shock Resistance								
Creep Properties								
Oxidation Resistance and Contamination					Slightly better than Ta			
Coating					No effect of coating on mechanical properties			
Experience with 45° brittleness					No tendency noted		No tendency noted	
Lamination tendency					No tendency noted		No tendency if properly rolled	
Ingot size	2"		3-1/2" Dia. x 6"		3-1/2" Dia. x 6"		8" Dia. x 24	5-1/2"
Sheet size	6 x 20		6 x 43 x 0.045		5 x 20 x 0.045		15 x 48 x 0.030	24 x 96 x 0.030
Status							Commercial	Commercial

10. PROPERTIES OF CANDIDATE TANTALUM ALLOYS

(As Presented at November 3, 1961, Meeting)

Battelle (10Hf - 5W)		Wah Chang (10W)		National Research Corporation (10W)		Westinghouse (8W - 2Hf)		Fansteel (10-1/4W -0.15Zr)		Stauffer (10W Modified)		Stauffer (Mark II)	
R. Annealed													
135	105	(200*)	98**	(180)		135		133					
130	98	197	90**	(160)		130		130					
15	25	4	12+**	4		15		2.7					
*Numbers in parentheses are estimates													
**Properties are stress-relieved													
400	3000	3590		2200 F	2500 F	3000 F	3500 F	2200	2500	3000	2400	3000	
39	18	(11)		67	22	15	7.5	85	54	20.5	39	18	
31	15.7	(8)		55	20	13	7.2	78	38	19.5	38	14	
55	51	(53)		4	22	25	37	15	26	60	19	71	
								At 2200 F and 19,000 psi, crept 0.068%/hr. with R. L. of >1.5 hrs. at 2600 F and 16,000, 1.75%/hr. & 9.8% hrs.					
2400 F		2600		2500 F		(2800 F) 25% aft. 1/2 hr. at 2730 F		2600		(2800 F)		(2600 F)	
<-320 F						<-320 F over 3T						More than 2T	
<-320 F				"Slightly above liquid- nitrogen temperature"		<-320 F				-190		(-70)	
2 - 4T Brittle		4T				0T 1.3T							
0.599 5414 ± 90						0.604		0.62					
27						28 at R. T.; 7 at 2400; 7 at 3000							
an Ta						None observed							
mechanical properties						None observed using							
no tendency noted						reverse bending							
no tendency if properly rolled													
1-1/2" Dia. x 6"		8" Dia. x 24		5-1/2" Dia.		3" Dia.		2" Dia.		5" Dia. x 40		5" Dia.	
x 20 x 0.045		15 x 48 x 0.030		24 x 96 x 0.030"		6 x 24 x 0.020"		6 x 20 x 0.030"		12 x 24 x 0.060"		4 x 20 x 0.060"	
		Commercial		Commercial						Commercial			

tending to resolve these questions should be acquired and reviewed by late Spring, 1962, with the possibility in mind of recommending support in a sheet rolling program at that time.

April 17, 1963 (see Table 11)

(1) The T-111 tantalum candidate (Ta-8W-2Hf) has been withdrawn by Westinghouse in favor of a modified T-111 (Ta-10W-2.5Hf-0.01C) composition. Ingots and sheets corresponding to the ground rules for pilot status have been produced of the modified alloy.

(2) The Westinghouse modified T-111 alloy met the Refractory Metals Alloy Requirements & Selection Subpanel property targets, excepting 3500⁰F tensile strength, and possessed a moderate but significant improvement in elevated-temperature strength over the commercial Ta-10W alloy. However, it was noted that many of the desired target properties were not determined, and that there was a discrepancy between the Westinghouse and AMRA creep-rupture-strength values.

(3) The Westinghouse modified T-111 alloy was recommended for uniform evaluation at AMRA, and if the Westinghouse data are confirmed, for a pre-production sheet rolling program.

(4) National Research Corporation provided data indicating that low-interstitial Ta-10W and Ta-8W-2Hf have comparable strength and ductility properties, indicating that the superiority noted in the early Westinghouse T-111 data was influenced by high-interstitial content.

(5) The Battelle-du Pont Air Force scale-up alloys, Ta-5W-2.5Mo and Ta-10W-2.5Mo, so far have not been produced in sheet sizes corresponding to the ground rules for pilot size, nor have properties corresponding to the Alloy Requirements & Selection Subpanel targets yet been determined. On the basis of limited information, it appears that the Ta-5W-2.5Mo alloy possesses no strength advantage over Ta-10W, and the higher strength Ta-10W-2.5Mo is not ductile in the welded condition.

(6) In general, the ARSS was disappointed that tantalum-alloy candidates greatly superior to Ta-10W have not been offered. It is noted,

TABLE 11. TANTALUM-ALLOY CANDIDATES

(As presented at April 17, 1963 Meeting)

Property	Target	Westinghouse				Battelle-Du Pont		National Research Corporation	
		Low-Int. T-111 (8W-2HF)	Mod. T-111 (9.6W-2.4HF-0.01C)	5W-2.5Mo	10W-2.6Mo	10W	8W-2HF		
Room-Temperature Tensile	(b) 15(a)	150-145-9							
Optimum, SR	(b) 15	150-145-9	--	--	--	111-107-15	113-98-20		
Recrystallized	(b) 15	84-67-31	110-105-25	100-86-35	112-105-25	--	--		
Elevated-Temperature Tensile									
2400 F	35-28-(b)	38-26-29	53-38-20	--	--	40-35-19	40-20-11		
2700 F	--	--	--	21-19-85	23-25-80	--	--		
3000 F	25-16-(b)	15.6-14.5-31	25-24-24	--	--	19-16-84	20-18-29		
3500 F	15-10-(b)	8.75-8.5-32	--	--	--	--	11-11-31		
Stress-Rupture Strength, ksi									
Temperature, F	2400 3000	2400	--	2700 3500	2400 3500	2400	2400		
10 Hr	(b) (b)	26	--	12 36	15.5 5.3	23	21		
50% Recrystallization Temperature, F (1 Hr)	(b)	2550	--	2400 (Ext)	2800 (Ext)	2400 2500	2400 2500		
Transition Temperature, F (4T)	-320	<-320(2T)	<-320(2T)	<-425	-300	<-320	--		
Room-Temperature Bend Ductility									
Base	1T	1T	<2T	0T	1T	0T	0T		
Weld	2T	2T(-320 F)	2.5T (-250 F)	2-12T	Brittle	1T	1T		

(a) Ultimate strength in ksi, 0.2% offset yield strength in ksi, and per cent elongation in 1 inch.

(b) To be furnished.

however, there may be an advantage in alloys containing reactive metal additions, like Ta-W-Hf, to serve as an internal getter in applications involving liquid metals.

(7) It is recommended that research and development programs calling for Westinghouse T-111 alloy should consider substitution of modified T-111 alloy.

May 21, 1964 (see Table 12)

(1) The Alloy Requirements & Selection Subpanel identified the General Electric 473 (Ta-7W-3Re) and Westinghouse T-222 (Ta-10W-2.5Hf-0.01C) alloys as the two outstanding tantalum alloys so far developed. Alloy development appears to have resulted in optimized compositions, and little future improvement can be expected in weldable-class tantalum alloys.

(2) The processing procedure for General Electric 473 appears to have been well worked out, and there appears to be no need for Government support on production-level processing. Because there are no known present or future requirements for high-strength tantalum sheet, no recommendation was made for a production sheet rolling program for Phase II and Phase III evaluations.

(3) Additional process development of Westinghouse T-222 is in progress for the Navy, which will answer questions about the feasibility of scale-up to large ingots. It is recommended that Westinghouse investigate the consolidation starting with tantalum powder in this contract.

Tungsten Alloys

November 30, 1962

(1) The Alloy Requirements & Selection Subpanel was impressed by the great progress that had been made in the quality of high-strength sheet alloys of tungsten by the arc-cast and the powder-metallurgy methods.

(2) Since it appears that those things which needed doing to permit further judgment of high-strength tungsten sheet were, indeed, being done, further action by the ARSS was deferred until Spring, 1963, at which time the status and progress of both arc-cast and powder-metallurgy alloys will be reviewed.

TABLE 12. COMPLIANCE OF TANTALUM-ALLOY CANDIDATES WITH MAB TARGETS

(Presented at May 21, 1964, Meeting)

Property	Target	G. E. 473 (TW-3Re)	Westinghouse T222 (9.6W-2.4Hf-0.01C) and ST222 (11.2W-2.8Hf-0.01C)
Room-Temperature Tensile			
Stress Relieved	$f_{tu}-f_{ty}-15^{(a)}$	158-151-3.5 (CW, L)	--
Recrystallized	$f_{tu}-f_{ty}-15^{(a)}$	106-94-22.5 (L)	113-111-24 (T222)
Elevated-Temperature Tensile			
2400 F	35-28- ^a	30.6-21.8-70 (2520 F)	(55-64)-(37-40)-(25-23)
3000 F	25-16- ^a	26-17.5-49 (3040 F)	(26-29)-(24-28)-(55-65)
3500 F	15-10- ^a	14-10.9-32 (3550 F)	(15-13.7)-(14.5-13.3)-(35-48)
Stress-Rupture Strength, ksi			
2400 F			
1 Hr	State	--	43-51
10 Hr	"	--	35-42
3000 F			
1 Hr	"	8.4 (3500 F)	18-19
10 Hr	"	5 (3500 F)	12-13
50% Recrystallization Temperature, F (1 Hr)	State	<2732	
Transition Temperature, F			
4T Bend	-320	--	<-320 F
Tensile	State	<-320	<-275 F to <-250 F
Notch-Sensitivity Ratio ^(b)	1.2	--	1.17 (-320 F)
Room-Temperature Bend Ductility			
Base	1T	<1T	<1.6T
Weld	2T	<1T	<1.6T
Density, g/cc	State	16.8	--

(a) Ultimate strength in ksi, 0.2% offset yield strength in ksi, and per cent elongation in 1 inch.

(b) Where a range is given, the first value corresponds to T222 and the second to ST222.

April 17, 1963 (see Table 13)

(1) The Sylvania "A" tungsten alloy passed the ground rules for pilot production, and the alloy appeared to be a most interesting candidate for high-strength tungsten sheet.

(2) The numerical data presented on Sylvania "A" alloy appeared very interesting, though many of the desired target properties were not determined.

(3) Sylvania "A" alloy was recommended for uniform evaluation at the Army Materials Research Agency, Watertown Arsenal.

(4) It is understood that Sylvania will release information about the composition of "A" alloy after filing patent applications, and that this information should be available within the next several months.

(5) The Alloy Requirements & Selection Subpanel wishes to defer final recommendation until more complete information from Sylvania and the AMRA uniform evaluation data become available. However, tentatively, the alloy is being considered for Sheet Rolling Panel support on a preproduction level, at the 18 x 36-inch size.

May 21, 1964

(1) Sylvania W-0.5Hf-0.02C powder-metallurgy tungsten alloy apparently is being commercialized by Sylvania. It was not presented as a candidate for an alloy-sheet-rolling program. No recommendation is made relative to sheet-rolling support.

(2) The Allied granule tungsten product appears to be an interesting dilute tungsten class of material, with improved recrystallization and grain-growth characteristics compared with unalloyed tungsten. The development has not yet met Sheet Rolling Panel ground rules. Allied was encouraged to extend their evaluation at least to the pilot-stage development and have properties checked against the dilute tungsten targets. The Alloy Requirements & Selection Subpanel also recommended that the current BuWeps program at Battelle on evaluation of various types of tungsten, including Allied, evaluate the Allied material in pilot-size sheet against Sheet Rolling Panel target properties.

TABLE 13. HIGH-STRENGTH TUNGSTEN-ALLOY CANDIDATES

(Presented at April 17, 1963 Meeting)

Property	Targets		Sylvania "A" (Composition not disclosed)	Climax (W-3Mo)
	Dilute Tungsten	High-Strength Tungsten		
Room Temperature Tensile Optimum	2(a)	(b)	--	--
Recrystallized	(b)	(b)	--	--
Elevated-Temperature Tensile				
3000 F	20	36-24-(b)	74-?-?	23.2
3500 F	10-7-(b)	27-18-(b)	60-?-? (3400 F)	9.1
4000 F	--	15-10-(b)	--	--
Stress-Rupture Strength, psi				
Temperature, F	3000	3000	3000	3000
10 Hr	(b)	(b)	28	11.2
50% Recrystallization Temperature, F (1 Hr)	State	3400	--	3000
Transition Temperature, F (4T)	300	300	300-350	250-500
Room-Temperature Bend Ductility				
Base	4T (300 F)	4T (300 F)	--	--
Weld	(b)	(b)	--	--

(a) Ultimate strength in psi, 0.2% offset yield strength in psi, and per cent elongation in 1 inch.

(b) To be furnished.

(3) Encouraging laboratory studies are in progress at Battelle and NASA-Lewis on ductile tungsten alloys containing 3 to 5 per cent rhenium, which show room-temperature ductility. The Alloy Requirements & Selection Subpanel will continue to review progress on this class of alloy.

July 13, 1965 (see Table 14)

(1) A very promising situation exists with regard to new tungsten alloys. Substantial progress has been made within the last 2 years.

(2) Further developments of both the high-strength and ductile classes should be encouraged, possibly combining the several alloying mechanisms.

(3) A selection of the most promising alloy or alloys could be made in the future. One reason a selection was not made at this time is that the properties of the various alloys constitute different combinations of virtues and liabilities - some emphasizing strength, others ductility, weldability, etc. Since firm requirements are not known now, a selection at this time would be premature.

(4) The selected alloy or alloys should be scaled up, at least at the pilot level, to demonstrate feasibility and to determine design data.

Specific comments regarding the alloys presented were as follows:

A. Ductile Class

NASA-Lewis (Electron-Beam-Melted W-2Re). This alloy has strength properties comparable with those of tungsten, but much superior low-temperature ductility and ease of fabrication.

General Electric LMCD (W-25Re). This alloy displays remarkable room-temperature strength and ductility (ultimate tensile strength 275 ksi, 14 per cent elongation). Welds relatively brittle and lacks stability needed in some nuclear applications.

General Electric NMPO (W-27Re-20Mo). This alloy has most of desirable characteristics of W-25Re, with somewhat lower melting point but superior low-temperature ductility, weld ductility, and elevated-temperature stability.

TABLE 14. SUMMARY OF ADVANCED TUNGSTEN ALLOY DEVELOPMENT

Classification	Organization	Candidate	Room Temp. Properties		Elevated Temp. Properties		Remarks
			Stress Relieved	Recrystallized	3000 F(b)	3500 F(c)	
Ductile W	NASA-Lewis	E. B. W-2Re	4T DBTT -75 F (No ductility in bar)	4T DBTT 425 F (No tensile data)	18-7-82	11-5-85	Comparable strength with tungsten, but better low-temperature ductility. R x temperature 2700 F. No weld data.
Ductile W	G.E.-LMCD	W-25Re	3T DBTT <-20 F 275-260-14(a)	3T DBTT <RT 190-165-12(a)	30-12-7	20-9-7	Loses R. T. ductility after long exposure ~4500 F. welds ductile at R. T. R x temp ~ same as W
Ductile W	G.E.-NMPO	W-27Re-20Mo	Ductile	Ductile	No data	No data	Maintains R. T. ductility after long exposure ~4500 F. welds ductile at R. T. M.P. ~5070 F. Stronger at elevated temperature than W-25Re
Ductile W	Battelle	W-5Re-2.2ThO ₂ (Doped)	4T DBTT 175 F 8T DBTT RT 179-158-12(a)	4T DBTT 257 F 146-135-4(a)	37-28-20(SR)	16-10-12(SR) 9-7-44(Rx)	Consolidated by gas-pressure bonding, no weld data, R x ~3270 F
Ductile, high-strength W	G. T. and E.	W-Re-(Hf/Zr)-C	4T DBTT 32-50 F No tensile data	No data	62-45-20(SR)	No data	Rx ~3100 F, no weld data or Rx properties
High-strength W	Westinghouse-Bloomfield	W-2ThO ₂ (Special K)	No data	No data	29-26-11(SR) (3350 F)	No data	Very very fine dispersion (500 Å) ThO ₂ Rx ~2900 F. stable after 1 hr. 4900 F. no sheet or weld data
High-strength W	NASA-Lewis	W-0.2Hf-0.17C	No data	No data	88-60-16 (As swaged)	62-57-17 (As swaged) 36-29-30 (Rx)	Rod data only, no weld data, Rx 4200 F, good fabrication char.
High-strength W	Climax	W-0.5Zr-0.05C	TT ~800 F No RT data	No data	79-7-12 (Swaged)	62-43-17 (Swaged) 42-24-25 (Rx)	Bar data only, Rx ~4000 F, poor fabrication char., no weld data.

TABLE 14. SUMMARY OF ADVANCED TUNGSTEN ALLOY DEVELOPMENT

Classification	Organization	Candidate	Room Temp. Properties		Elevated Temp. Properties		Remarks
			Stress Relieved	Recrystallized	3000 F(b)	3500 F(c)	
High-strength W	T. R. W.	W-20Ta-12Mo	TT~1500 F	No data	72-67-12 (Extruded) 64-44-60 (Rx)	29-28-35 (Rx)	Poor fabrication char., Rx~3300 F. Very poor fabrication char., R
		W-12Cb-0.29V- 0.12Zr-0.07C	TT~2500 F	No data	85-?-25 (Extruded)	45-?-30 (Extruded)	Rx~>4100 F

(a) Gives UTS in ksi, 0.2% offset yield strength, and % elongation in 1 inch.

(b) For comparison, at 3000 F unalloyed tungsten has ultimate strength 13 ksi and 0.2% offset yield 6.5 ksi.

(c) For comparison, at 3500 F unalloyed tungsten has ultimate strength 9 ksi and 0.2% offset yield 3.5 ksi.

Battelle (Doped W-5Re-2.2ThO₂). This alloy has high room-temperature strength and ductility. Weld ductility is low.

Sylvania (W-Re-Hf or W-Re-Zr). This alloy shows a unique combination of high-temperature strength and low-temperature ductility. Weld data lacking.

Linde-Philco (Single-Crystal Tungsten). Not much promise was seen for the single-crystal route for sheet applications.

B. High-Strength Class

Westinghouse (W-2ThO₂). This alloy has outstanding stability of a very fine ThO₂ dispersion. Sheet data lacking.

NASA Lewis (W-0.2Hf-0.17C). This alloy has good fabricability and very high elevated-temperature strength.

Climax (W-0.5Zr-0.05C). This alloy is similar to the above, but Climax has experienced much greater fabrication difficulty.

TRW (W-20Ta-12Mo and W-12Cb-0.3V-0.12Zr-0.07C). These alloys possess poorer fabricability and higher ductile-brittle transition temperatures, relative to dilute tungsten-base alloys of comparable strength.

APPENDIX III

EVALUATION OF CANDIDATE REFRACTORY METAL SHEET ALLOYS
BY THE U. S. ARMY MATERIALS RESEARCH AGENCY

In December 1959, shortly after establishment of the Materials Advisory Board Panel for Refractory Metals Sheet Program, the U. S. Army representative remarked that experience during earlier titanium sheet alloy development had demonstrated that selection of candidate alloys and recommendation of these for Government support should not depend upon data supplied solely by the producers. He suggested that an independent laboratory test samples of such alloys and certify those properties which the Alloy Requirements & Selection Subpanel might elect. The following advantages of such action were foreseen:

1. Sheet samples of the various candidate alloys could be evaluated in identical fashion, using methods recommended by the Subpanel on Standardization of Test Methods, so that direct comparison of their respective properties would be possible.
2. Complete characterization of each candidate, to the extent required in "ground rules" established by the Alloy Requirements & Selection Subpanel, would be accomplished.
3. Selection of more promising alloys for which to recommend government support would be facilitated by confirming and clarifying these critical properties forming the bases for selection.

At that time acquisition of property information for candidate alloys as required by the ARSS was difficult. Procedures for elevated temperature testing of refractory metal sheet had not been adequately developed, standardization of methods was lacking, both sample material and test equipment were costly. For these reasons reported properties were not infrequently suspect and complete characterization of candidate alloys in accordance with ARSS requirements was seldom achieved by the producer. As a result of this situation, the Panel recommended that a single qualified laboratory test all candidate sheet alloys as part of the ARSS evaluation procedure. The U.S.

Army Materials Research Agency volunteered to provide such technical personnel, equipment and supporting funds as would be required for this purpose.

It is not claimed that AMRA findings were more accurate than those of any other laboratory. However, the concept of a single laboratory to provide property data enabling comparison of candidate alloys does not require that these data be precisely correct, provided that they are of consistent accuracy. The excellent consistency of the AMRA data can be judged by inspecting AMRA Technical Reports Nos. 64-16 and 65-25.

A continuing problem in AMRA evaluation of candidates was the transient nature of these developmental alloys. Since evaluation of a sample commonly required a period of months, during which time development of the alloy continued at the producer's laboratory, it was not unusual to find newly acquired data obsolete at time of publication. Indeed, during the most active period of the program, influx of candidates for evaluation (and continued development of these same candidates at a high rate of effort) exceeded the capability of the AMRA group to keep up with the increasing workload. Inasmuch as the AMRA property data proved most useful in those instances where it was available as a basis for selection of competitive candidates, this experience should be recognized. Future Government-supported alloy development programs, wherein essential testing procedures are both difficult and nonstandardized, and wherein comparable property data are necessary as the means for selection, should make adequate provision to properly accomplish this evaluation service.

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13 ABSTRACT <p>The genesis, method of operation, and accomplishments of the Refractory Metals Sheet Rolling Panel are described in the report of the main Panel, along with reflections on the conduct of such a program and recommendations for future activities in this field.</p> <p>Summary reports of the eleven subpanels and one special <u>ad hoc</u> subpanel are included in the body of the report. Longer discussions of activities of the Subpanel on Alloy Requirements & Selection constitute an appendix to the report.</p>		

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